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# Life Improvement of Alloys in Heat Treatment Furnaces and Fixtures

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# Life Improvement of Alloys
# in Heat Treatment Furnaces and Fixtures

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## Abstract

Heat treatment furnace components and part holding fixtures are comprised of expensive heat-resistant alloys that are typically consumed in industry in less than three years, requiring frequent replacement. This project investigates how to improve the life of materials used in heat treatment applications through failure analysis of the current materials used in industry, and by researching new materials and diffusion treatments. Determined through multiple visuals, constituent measurements, and micro-hardness tests a sample racking post that had been used in industry, made of HT alloy, failed due to carburization.

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## 1.0 Introduction

The Center for Heat Treating Excellence (CHTE) is always searching for ways to improve and advance heat treatment technology. There are a variety of different heat treatment furnaces in industry, all of which are designed to heat treat a plethora of various alloys at specific temperature ranges, for a specified amount of time, and in a gaseous atmosphere suitable improve upon given alloyed parts. The furnaces that encase the heat treating process undergo daily stresses due to the extreme changes in environment and cyclic changes in temperature. These furnaces are made up of an assortment of different materials; both metal alloys and ceramic composites are used to create furnace parts and fixtures. It has been brought to the attention of CHTE that the materials used in furnace parts and fixtures are expensive relative to their functional life span. This project looks to begin to improve the life of materials that are being used for furnace parts and fixtures, and to investigate newer material technologies.

## 2.0 Literature Review

Materials currently being used for parts in heat treatment furnaces and fixtures are made of heat-resistant materials. These materials need to be able to constantly operate at 540-1200°C (1000-2200°F) [1]. Based on these requirements, it is important to understand that selecting appropriate alloys to use in furnace applications does not depend on the room temperature mechanical properties except to make ensure the alloy is of good quality [1]. At elevated temperatures the alloys are subject to greater stresses under given loads and are more susceptible to undergo changes in deformation.

In general selecting the right alloy for use in a given high temperature application, specifically in furnaces, is dependent upon many factors and operating conditions. Operating costs and related mechanical and physical properties of an alloy to consider are outlined in the following table (Table 1) gathered from an article distributed by the Nickel Development Institute.

Table 1: Selecting the right alloy for use in a given application[2]

| Operating Conditions | Related Property |
| --- | --- |
| **1. Anticipated service and maximum temperature of operation** | Short-time tensile properties<br>Creep Strength<br>Stress-rupture properties<br>Hot Ductility |
| **2. Type and size of maximum load** | Short-time tensile properties<br>Creep Strength<br>Stress-rupture properties<br>Hot Ductility |
| **3. Temperature cycling**<br>**a. Range of temperature cycling**<br>**b. Frequency of temperature cycling**<br>**c. Rate of temperature Change** | Thermal fatigue properties |
| **4. Type of atmosphere or other corrosive conditions** | Oxidation resistance<br>Carburization resistance<br>Sulfidation resistance<br>Surface stability |
| **5. Size and shape of part** | Temperature gradients |
| **6. Further processing, such as welding and machining** | Fabrication data |
| **7. Abrasive or wear conditions** | - |
| **8. Cost** | - |
| **9. Ease of replacement** | - |

Both cast and wrought alloys are used for parts and fixtures (see Table 2, Table 3, and Table 4, taken from ASM Heat Treating handbook volume 4, for lists of typical alloys used for furnace parts and fixtures). Iron-chromium-nickel alloys, iron-nickel-chromium alloys, and nickel-base alloys are most commonly used in heat-treating furnaces [1]. Nickel increases the alloys strength and toughness at high temperatures, whereas chromium creates good oxidation resistance [1]. Cobalt-base alloys are another type of alloy that is seen in heat-treatment furnaces, although they are of good quality they are generally the more expensive option [1]. Other elements seen in these alloys include primarily carbon, which also increases an alloys strength, silicon and manganese [1].

**Table 2: Heat-resistant cast alloys typically used and their properties at high-temperatures [1]**

| | Approximate composition, % | | | | Creep stress to produce 1% creep in 10,000 h | Stress to rupture in 10,000 h |
|---|---|---|---|---|---|---|
| Grade | C | Cr | Ni | °C | Mpa | Mpa |
| Iron-chromium-nickel alloys | | | | | | |
| HF | 0.2-0.4 | 19-23 | 9 to 12 | 650 | 124 | 114 |
| | | | | 760 | 47 | 42 |
| | | | | 870 | 27 | 19 |
| | | | | 980 | | |
| HH | 0.2-0.5 | 24-28 | 11 to 14 | 650 | 124 | 97 |
| | | | | 760 | 43 | 33 |
| | | | | 870 | 27 | 15 |
| | | | | 980 | 14 | 6 |
| HK | 0.2-0.6 | 24-28 | 18-22 | 650 | | |
| | | | | 760 | 70 | 61 |
| | | | | 870 | 41 | 26 |
| | | | | 980 | 17 | 12 |
| Iron-nickel-chromium alloys | | | | | | |
| HN | 0.2-0.5 | 19-23 | 23-27 | 650 | | |
| | | | | 760 | | |
| | | | | 870 | 43 | 33 |
| | | | | 980 | 16 | 14 |
| HT | 0.35-0.75 | 15-19 | 33-37 | 650 | | |
| | | | | 760 | 55 | 58 |
| | | | | 870 | 31 | 26 |
| | | | | 980 | 14 | 12 |
| HU | 0.35-0.75 | 17-21 | 37-41 | 650 | | |
| | | | | 760 | 59 | |
| | | | | 870 | 34 | 23 |
| | | | | 980 | 15 | 12 |
| HX | 0.35-0.75 | 15-19 | 64-68 | 650 | | |
| | | | | 760 | 44 | |
| | | | | 870 | 22 | |
| | | | | 980 | 11 | |

**Table 3: Heat-resistant wrought alloys typically used and their properties at high-temperatures [1]**

| | | Approximate composition, & | | | | Creep stress to produce 1% creep in 10,000 h | Stress to rupture in 10,000 h |
|---|---|---|---|---|---|---|---|
| Grade | | C | Cr | Ni | °C | Mpa | Mpa |
| ron-chromium-nickel alloy | | | | | | | |
| 306S | | 0.08 | 22-24 | 15 | 650 | 48 | |
| | | | | | 760 | 14 | |
| | | | | | 870 | 3 | 10 |
| | | | | | 980 | | 3 |
| 310S | | 0.06 | 24-26 | 19-22 | 650 | 63 | |
| | | | | | 760 | 17 | |
| | | | | | 870 | 9 | 13.5 |
| | | | | | 980 | | 4 |
| Iron-nickel-chromium allo | | | | | | | |
| RA 330 | | 0.08 | 17-20 | 34-37 | 760 | 25 | 30 |
| | | | | | 870 | 13 | 12 |
| | | | | | 980 | 3.5 | 4.5 |
| RA 330 HC | | 0.4 | 17-22 | 34-37 | 760 | 47 | 54 |
| | | | | | 870 | 18 | 18 |
| | | | | | 980 | 5 | 5 |
| RA 333 | | 0.08 | 24-27 | 44-47 | 760 | 43 | 65 |
| | | | | | 870 | 21 | 21 |
| | | | | | 980 | 6 | 7 |
| Incoloy 800 | | 0.1 | 19-23 | 30-35 | 760 | 19 | 23 |
| | | | | | 870 | 4 | 12 |
| | | | | | 980 | 1 | 6 |
| Incoloy 802 | | 0.2-0.5 | 19-23 | 30-35 | 760 | 83 | 79 |
| | | | | | 870 | 30 | 33 |
| | | | | | 980 | 8 | 11.5 |
| Nickel-based alloys | | | | | | | |
| Inconel 600 | | 0.15 | 14-17 | 72 | 760 | 28 | 41 |
| | | | | | 870 | 14 | 16 |
| | | | | | 980 | 4 | 8 |
| Inconel 601 | | 0.1 | 21-25 | 58-63 | 760 | 28 | 42 |
| | | | | | 870 | 14 | 19 |
| | | | | | 980 | 5.5 | 8 |

Table 4: Recently developed heat-resistant wrought alloys typically used and their properties at high-temperatures [1]

| | Composition wt% | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Alloy | Fe | Ni | Co | Cr | Mo | W | C | Major Characteristics |
| 253 MA 9a0 | Bal | 11 | | 21 | | | 0.08 | Oxidation resistance |
| RA85H(b) | Bal | 15.4 | | 18.5 | | | 0.2 | Carburization resistance |
| FeCralloyA(c ) | Bal | | | 15.8 | | | 0.03 | Oxidation resistance |
| HR-120(d) | Bal | 37 | | 25 | | | 0.05 | Creep-rupture strength |
| 556(d) | Bal | 20 | 18 | 22 | 3 | 2.5 | 0.1 | Creep-rupture strength |
| HR-160(d) | 2 | Bal | 29 | 28 | | | 0.05 | Sulfidation resistance |
| 214(d) | 3 | Bal | | 16 | | | 0.05 | Oxidation resistance |
| 230(d) | | Bal | | 22 | 2 | 14 | 0.1 | Creep-rupture strength/oxidation resistance |
| Inconel 617 (c ) | 1.5 | Bal | 12.5 | 22 | 9 | | 0.07 | Creep-rupture strength/oxidation resistance |
| Incoloy MA 956 (c ) | Bal | | | 20 | | | | Creep-rupture strength/oxidation resistance |

Whether cast or wrought alloys are used in in heat-treatment furnaces or fixtures is dependent upon the specified design and operating conditions of the equipment. Determining factors include production rates or workloads, the rate of temperature change, whether or not quenching is involved, the desired service life of the equipment and the furnace atmosphere [1]. Both cast and wrought alloys bring certain advantages and disadvantages to the table to consider. Wrought alloys can be formed in practically any size, they have high thermal-fatigue resistance, carburization resistance, and are made with fewer defects internally and externally [1]. Castings can be found at lower costs, which can be fabricated in more elaborate shapes than most wrought alloys and can be found in a wide variety of compositions. Table 5 below from the ASM Heat Treating handbook presents certain cast and wrought alloys that are currently used for specific parts in heat treating applications.

**Table 5: Typical materials used for parts and fixtures for carburizing and carbonitriding furnaces [1]**

| | 815-1010°C | |
|---|---|---|
| Part | Wrought | Cast |
| Retorts, muffles, radiant tubes, structural parts | RA 330 | HK |
| | 800H/800/HT | HT |
| | HR-120 | HU |
| | 600 | HX |
| | 601 | |
| | 617 | |
| | X | |
| | 214 | |
| | 556 | |
| | 230 | |
| Pier caps, rails | RA 330 | HT |
| | 800H/800/HT | |
| | HR-120 | |
| | 600 | |
| | 601 | |
| Trays, baskets, fixtures | RA85H | |
| | RA330 | |
| | 800H/800/HT | HT |
| | HR-120 | HT (Nb) |
| | 300 | HU |
| | 601 | HU (Nb) |
| | 617 | HX |
| | X | |
| | 556 | |
| | 214 | |
| | 230 | |

## 2.1. Types of furnaces and heat treating processes

It is crucial to consider the different types of heat treating furnaces, and heat treating processes to understand what material properties are of most importance and the type of atmospheres that alloys may encounter in their lifetime.

### 2.1.1. Gas carburizing

Gas carburizing furnaces add carbon from the atmosphere to the steel surface at 850-980°C (1600-1800°F) [3, 4]. A low-carbon steel part will result with a carbon gradient which produces a strong and hardened surface layer[1]. In most gas carburizing furnaces, an endothermic gas is created by mixing air with a natural gas (or propane) in a gas cracker (or a separate retort furnace) over a heated catalyst to form a mixture of 40% nitrogen, 20% carbon monoxide, and 40% hydrogen [3, 4]. There are however, several different classes of gas carburizing furnaces, whose processes slightly vary and atmosphere compositions change. Carbon monoxide is not the only type of gaseous hydrocarbon that can be used for gas carburization; propane ($C_3H_3$) and butane ($C_4H_{10}$) can be used as well [1]. The carbon monoxide concentrations inside of the furnace are constantly monitored to ensure that there is a proper carbon potential so that the carbon will diffuse into the parts[3]. In many cases the surface carbon content of the work-piece may increase from 0.1% to 1.2%, after which the work-piece is quenched at the desired speed and temperatures to obtain a specified hardness [3, 4]. It is also important to control these parameters as the furnace parts and fixtures themselves are also subject to reach their solubility limits of carbon in austenite at their surfaces[1]. An overly concentrated atmosphere of carbon would subject the furnace parts and fixtures to unnecessary and unwanted carburization; it would decrease the integrity of the parts and fixtures over a period of time and cause unwanted failures.

There are two types of gas furnaces that can be used; batch (Figure 1, Figure 2) and continuous furnaces, both of which can vary widely in construction [1]. In batch furnaces the parts are loaded and unloaded as one unit at a specific time, whereas in continuous furnaces parts are continually entering and exiting the furnace on a unit such as a conveyor belt, which may be more desirable for production rates [1].

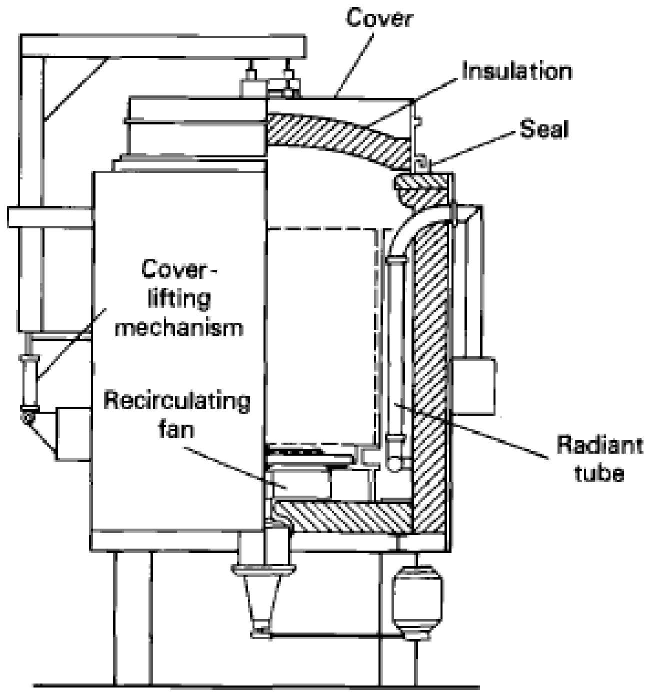



**Figure 1: Pit batch carburizing furnace schematic [1]**

In either case there are three main variables that must be properly controlled for a successful outcome; temperature, time and concentration of carbon in the atmosphere [1].

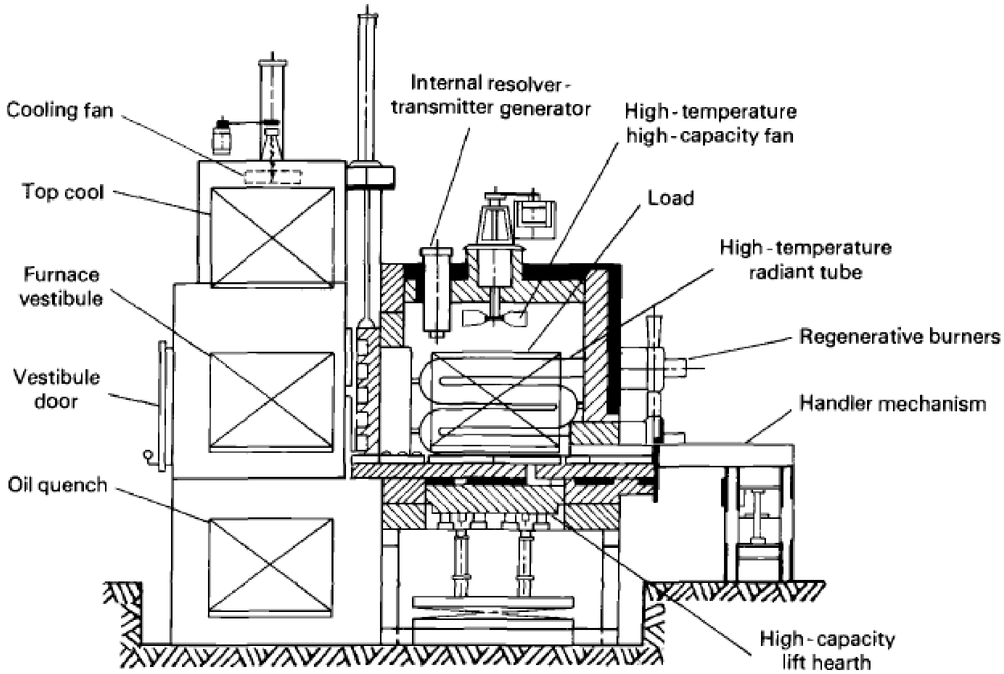



**Figure 2: Integral horizontal batch carburizing furnace [1]**

### 2.1.2. Vacuum carburizing

Vacuum carburizing furnaces are typically used at a higher temperature compared to that of gas carburizing furnaces, which can reduce cycle times significantly and result in a more productive process [1]. Vacuum carburizing is also performed at pressures well below atmospheric pressure [5]. However, with such low cycle times and high temperatures the types of alloys being used for furnace parts and fixtures must be reconsidered, since the alloys used within a vacuum furnace need to have an even higher heat-resistance than those used in gas furnaces. Despite the reduced cycle times, vacuum carburizing furnaces can maintain an effective carbon gradient on the surface of the part. Although it is difficult to obtain a smaller gradient with this process, grain refining is necessary and hard to maintain in a short period of time at high temperatures [1]. These furnaces operate at 900 - 1040°C (1650 - 1900°F)[1]. Vacuum carburizing furnaces are beneficial not only for reducing cycle times, but they also bring a very repeatable process to the table as the process is more easily controllable and leaves little time for intergranular oxidation to occur in parts, which causes a decrease in mechanical properties [6].

### 2.1.3. Carbonitriding

Carbonitriding is a form of gas carburization where ammonia is introduced into an atmosphere similar of gas carburization which adds nitrogen to the surface of the carburized part as it is being produced [1]. Nascent nitrogen is formed at the surface, nitrogen then diffuses into the steel with the carbon and creates a thin case comparative to that of gas carburizing [1]. Carbonitriding is generally performed at a lower temperature for a shorter period of time than gas carburizing and this is why a thin case is created [1]. In general a carbonitrided case has a greater hardenability than that of a carburized case and therefore has good wear-resistance [1].

## 2.2. Primary failure modes of furnace parts and fixtures

Literature on noted and possible primary failure modes of furnace parts and fixtures was read and analyzed to understand why alloys are failing at the rate that they are and how the problem can possibly be remediated.

### 2.2.1. Metal Dusting

Metal dusting is a form of extreme carburization seen in carburizing atmospheres in which metallic components degrade into metal powder (graphite or coke)[7, 8]. It occurs in iron, cobalt, and nickel-base alloys[7]. Metal dusting occurs at 430-900°C (800-1650°F), when the carbon activity is greater than one ($a_c$>1). It happens when oxygen levels are low and when methane, propane, ethane or any other hydrocarbons are present [8]. The maximum rate of metal dusting attack occurs at 600-706°C (112-1292°F) [8].

When an iron base alloy becomes over saturated with carbon at a high temperature, cementite ($Fe_3C$) forms on the surface of the alloy. Graphite then forms on top of the cementite and the carbon activity where the cementite is in contact with graphite decreases to one. The cementite is then unstable and thus decomposes into iron and carbon. The iron migrates into the graphite layers to form iron particles [8]. Figure 3 below shows a schematic of how metal dusting occurs over time.

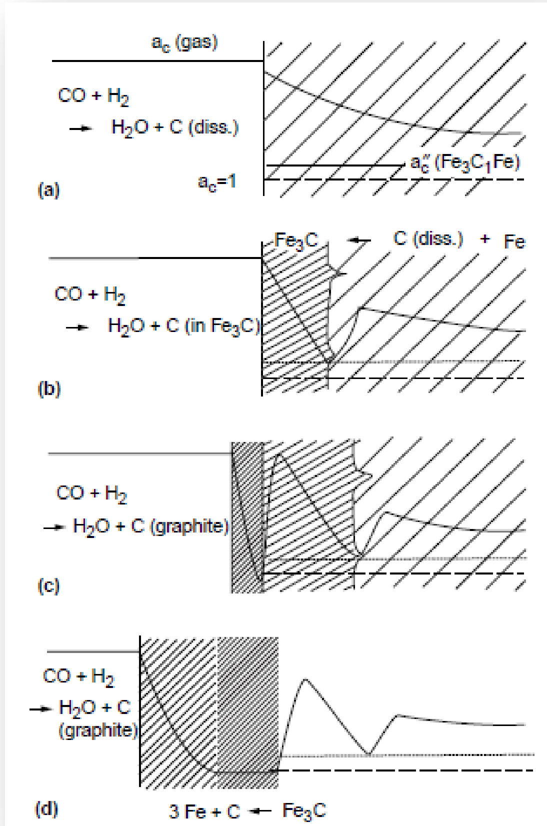



**Figure 3: Metal dusting schematic of Fe-alloy [8]**

Metal dusting of iron-alloys or low-alloy steels results in pitting or thinning of material and also the formation of internal carbides in the matrix of the attacked material [8]. Internal carbides cause embrittlement, a decrease in the materials ductility and creep-rupture strengths, and a loss in most basic material properties. Metal dusting also can occur in low-nickel alloys, however in these alloys $M_3C$ particles are formed at the surface of the alloy, known as 'coke' [8].

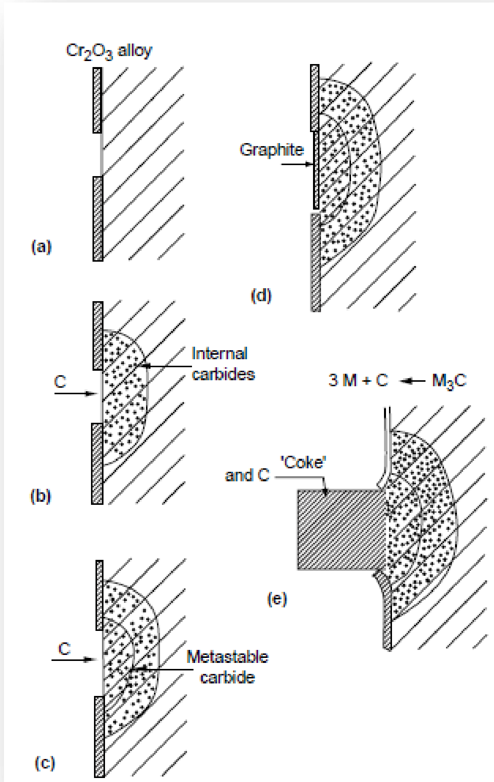



**Figure 4: Metal dusting schematic of a low-nickel alloy [8]**

Metal dusting is most commonly seen in petrochemical processes; however it is also seen in the heat treating industry. Refractory anchors and fan housing assemblies are two components of heat treatment furnaces that have exhibited degradation due to metal dusting [8]. Alloys used in heat treatment furnaces are most subject to this failure mode include 310, Ni-base alloys (alloys X and 333) and iron-base alloys (multimet alloys (N-155)) [8]. Metal dusting does not normally occur in heat treatment furnaces for at least a year after constant service and is most abundantly found in areas where gases are allowed to become stagnant in the furnaces [8].

Nickel base alloys with a high chromium and aluminum content are able to resist the effects of metal dusting fairly well. Nickel generally helps reduce the effects of carburization [2]. The chromium oxide scale acts as an inhibitor to metal dusting, therefore the higher the chromium concentration at the surface of the metal the faster the formation of protective scale [8]. Aluminum also produces $Al_2O_3$ scale that prevents metal dusting, which is better than most chromia formers ($Cr_2O_3$) [8]. High silicon content in nickel-base alloys is also advantageous because in carburizing atmospheres a produced $SiO_2$ scale is impervious to carbon [8]. However, one must consider the weldability of an alloy, as when too much silicon is introduced into an alloy the alloy's weldability will decrease. Sulfur rich atmospheres retard metal dusting attack as it segregates to the surface. Some heat treating processes will inject sulfur compounds (50-100ppm) into the gas stream to increase sulfur content [8]. Any surface machined alloy will also help retard metal dusting as the thin cold worked layer on the part surface creates a high density of dislocations, which provide paths for preventing the formation of oxide scale. Fine grained structures are also better for resisting metal dusting as their grain boundaries provide a fast diffusion path for chromium to reach the surface [8]. Figure 5 below graphically demonstrates the benefits of fine grains versus coarse grains when it comes to metal dusting attack.

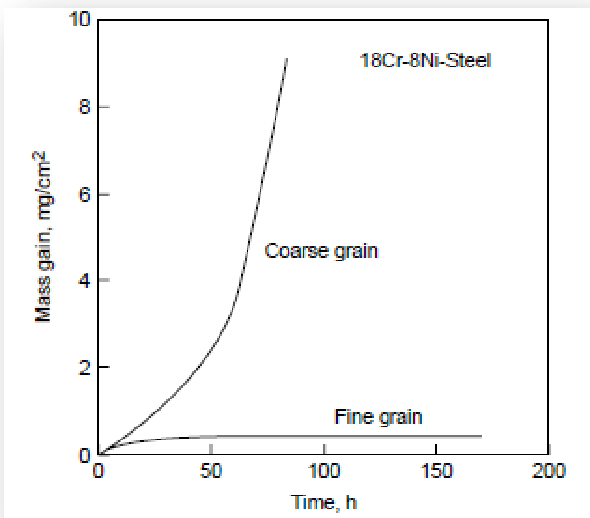



**Figure 5: Experiment results conclude that fine grained 18Cr-8Ni-steel does not lose nearly as much in mass due to metal dusting over a period of time as coarse grain 18Cr-8Ni-steel [8]**

### 2.2.2. Thermal Fatigue

Thermal fatigue is another failure mode of furnace parts and fixtures. Furnaces are constantly heated to high temperatures and reduced or shut off to lower temperatures. Fixtures are also often heated to these temperatures and then rapidly quenched, enduring a number of thermal cycles. Thermal fatigue is caused by this rapid heating and cooling [9]. This creates a large temperature gradient between the inner core and outer shell of the material [9]. Thus causes stresses as the part contracts and expands which then leads to failure of the part, warping, plastic deformation or micro- and/or macro-cracking of the part [9]. Because of thermal fatigue the life expectancy of trays and fixtures are commonly measured in the number of cycles they can withstand rather than the hours they are in use [1]. Conveyor chains, belts and any quenching fixtures are also regularly exposed to thermal shock [1]. Figure 9 below shows the effects of thermal fatigue on a grate used in a furnace.

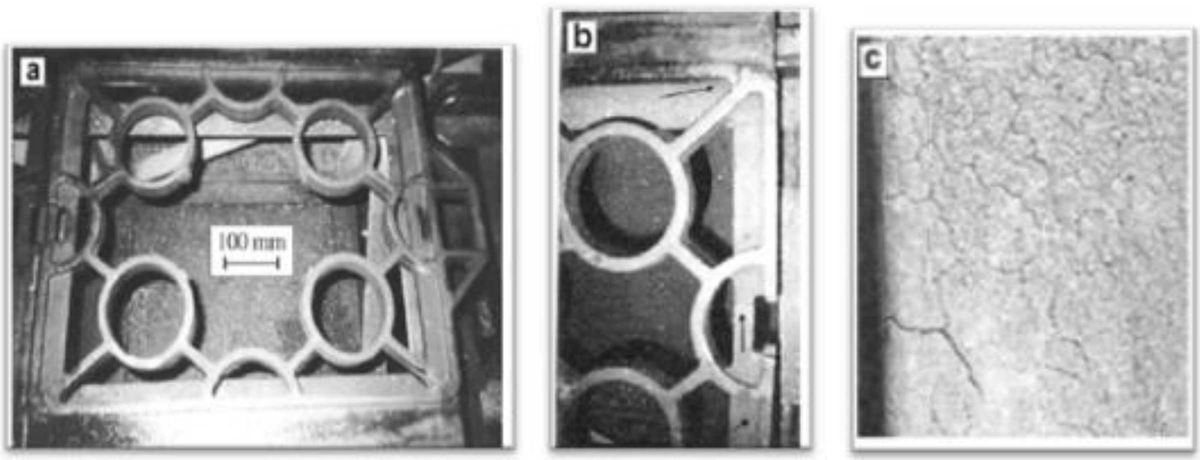



**Figure 6: (a) General view of a grate used in a furnace made of G-X40NiCrSi35-17, (b) places that exhibited thermal fatigue, (c) macro-cracks and deformation of the walls can be seen [9].**

### 2.2.3. Creep

Creep is also seen in furnace parts and fixtures used for heat treating as it is a time-dependent failure mode that causes deformation at high-temperatures and constant stresses. In furnace parts a creep rate of 0.0001% per hour is said to be satisfactory, however this does not mean that for every 10,000 hours 1% creep will withhold without failure for an acceptable heat resistant alloy [2]. Figure 7 shows a scanning electron microscope (SEM) image of a tube made of HP40 that failed due to creep.

Nickel-base alloys are advantageous in furnace applications as they have good creep-rupture strengths, as well as oxidation resistance [1].

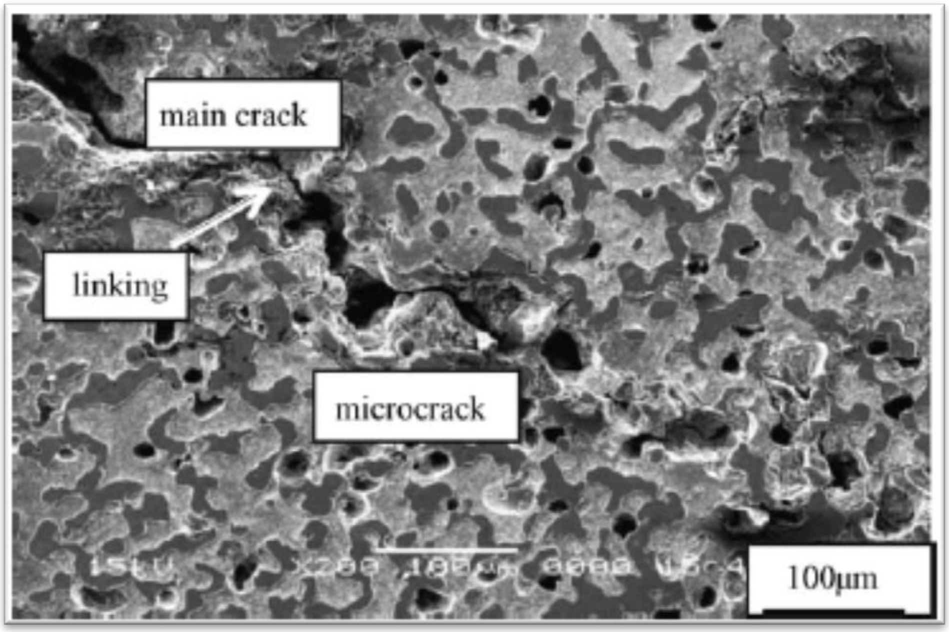



Figure 7: SEM image of crack tip on the surface of a failed tube that was used in a carburizing furnace made of HP40. Can see cracking along the austenite grains which has resulted from the connection of creep cavities [10].

## 2.3. Possible new materials to be considered

As research advances the possibility for using completely new materials, including composites may be a great alternative for heat treatment applications.

### 2.3.1. Carbon-carbon composites

Carbon-carbon composites were originally designed for use in aerospace applications, but have recently been considered for use in furnace fixtures as well. Carbon-carbon composites have great resistance to thermal fatigue and thermal shock, and have great strength [11]. Carbon-carbon composites are light weight, low in density, high modulus, a high fracture toughness, and good creep resistance [12, 13]. They also have a low specific heat which along with its strengths allows for a reduction in the amount of cycle times [11]. Another problem with metallic fixtures is the catastrophic propagation of cracks. With carbon-carbon composites cracks do not occur [11]. As seen in Figure 8 as temperatures increase, unlike most high-temperature alloys and materials, the UTS of carbon-carbon composites actually increases [14].

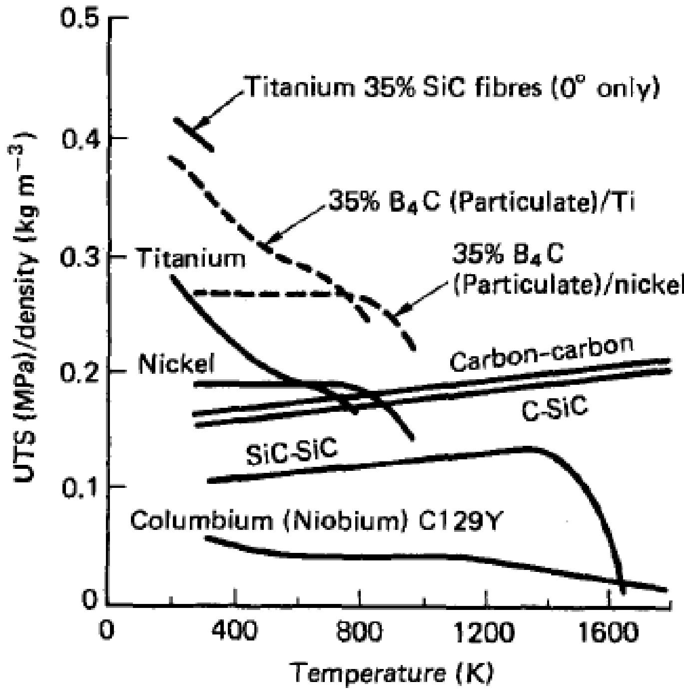



**Figure 8: Ultimate tensile strength comparison of several high-temperature materials with respect to temperature[14]**

Carbon Composites, INC. out of Leominster, MA, is already making electrodes, vacuum port penetration protectors, cylindrical liners, and disks out of carbon-carbon composites for vacuum furnaces [11].

### 2.3.2. Nickel aluminides

Nickel aluminides may have potential in furnace applications. Nickel aluminides have a very high carburization resistance and oxidation resistance at high temperatures (even above 1000°C) because of the formation of alumina[15]. Nickel aluminides are also advantageous as they have a high fatigue resistance, great tensile, compressive and creep strengths at high temperatures, and good wear resistance [15]. In one study the yield strength and ultimate tensile strength (UTS) of two nickel aluminides were plotted against that of HU, a common cast alloy used for furnace applications [15]. As seen in Figure 9 nickel aluminides present an increase in yield strength at higher temperatures compared to HU. Also, when examining ultimate tensile strength given graph (b) in Figure 9 it is clear that the nickel aluminides exhibit a better UTS. With these results one may be able to draw the conclusion the nickel aluminides have a greater potential in furnace applications than alloys such as HU, however cost must be taken into account as well. Nickel aluminides may provide a longer life than HU would because of its higher tensile and creep strengths as well as it's resistance to thermal fatigue [15].

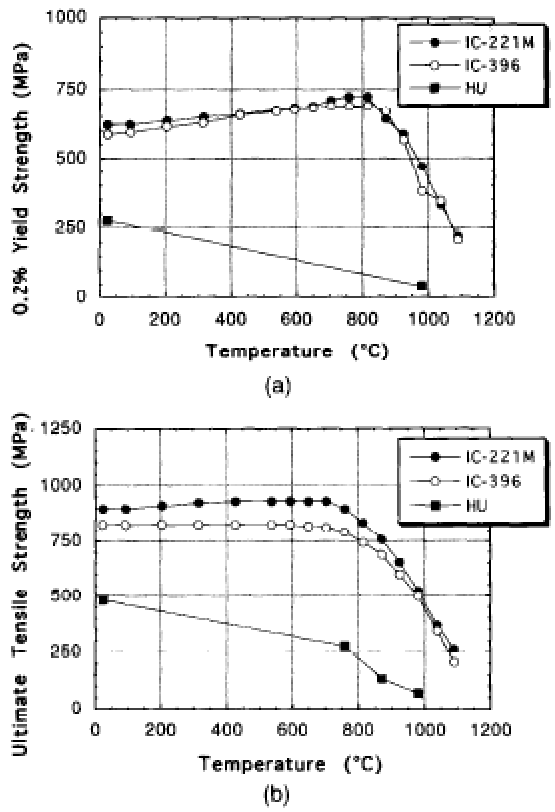



Figure 9: Nickel aluminides graphically compared to HU alloy [15]

### 2.3.3. Silicon-silicon carbide composites

Silicon-silicon carbide composites can also be used in applications for carburizing heat treatment furnaces, such as in radiant tubes [1]. The average life of these new tubes under continuous use is somewhere around 16 months whereas the life expectancy of the more commonly used mullite tubes is about a month [1, 4]. These composites provide clear advantages over alloys currently used for radiant tubes. Figure 10 shows a silicon-silicon carbide composite radiant tube after a compression creep test versus a commonly used Ni-Cr-Fe alloy radiant tube.

**Figure 10: Silicon carbide-silicon carbide composite tube seen on the left after 360hrs and Ni-Cr-Fe alloy seen on the right after less than 1 hour of high temperature (1350°C) creep testing of radiant tubes**

## 2.4. Diffusion related processes to improve lifetime of alloys

The threats to lifetime of alloys in furnaces and fixtures include: high temperature corrosion, high temperature oxidation, and carburizing atmosphere etc. To improve the life of alloys, coatings are widely used to increase the corrosion, oxidation and carburizing resistance. In general, there are two types of processes to make coatings [16]. One is to form the coating by changing the composition of the surface layer with diffusion and the other involves depositing additional layers on the surface of the parts. In this project, aluminizing consumed the main focus while for the time being silicon rich coatings were not considered.

### 2.4.1. Aluminizing

Aluminizing is based on the formation of intermetallic compounds of the nickel-aluminum ( Ni-Al) system that act as a reservoir of aluminum for maintaining a protective aluminum oxide ($Al_2O_3$) scale on the material surface during high temperature service [17]. Aluminizing is mainly done by the pack cementation.

Figure 11 shows the schematic of a pack aluminizing retort [18]. Components to be coated are packed by a powder mixture in a sealed retort. The pack mixture is composed of a donor alloy, an activator and an inert filler material. The donor is aluminum, which provides the coating element source. The activator is a halide salt to transport aluminum from the pack to the component. The most used inert filler material is alumina to prevent pack sintering during the process.

Recent developments have focused on gas-phase, Chemical Vapor Deposition (CVD), processes. [3] Figure 13 presents a gas-phase CVD retort [18]. Pellets instead of powder can be used in this method to avoid the usage of inert filler. Therefore, the cost of process is reduced. The basic mechanism of gas-phase CVD is the same as that of pack aluminizing, however the kinetics of Vapor Phase Aluminizing (VPA) is not as well-known as pack aluminizing [17].

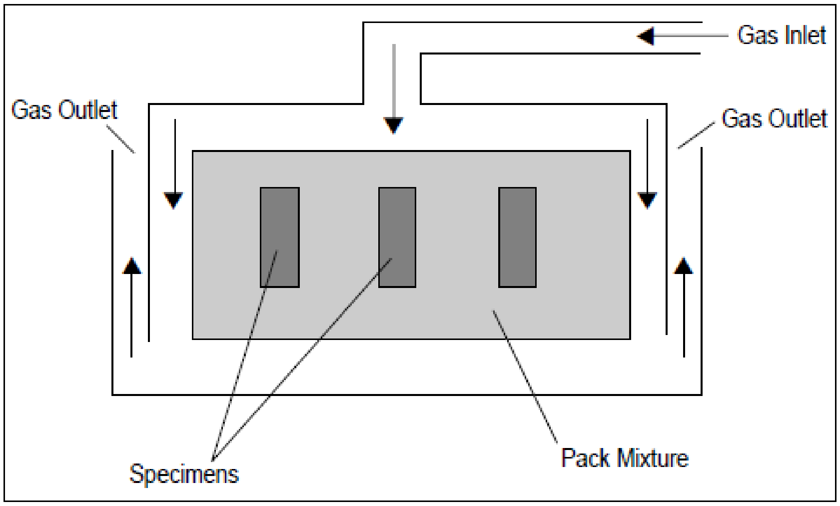



**Figure 12: A pack aluminizing retort [18]**

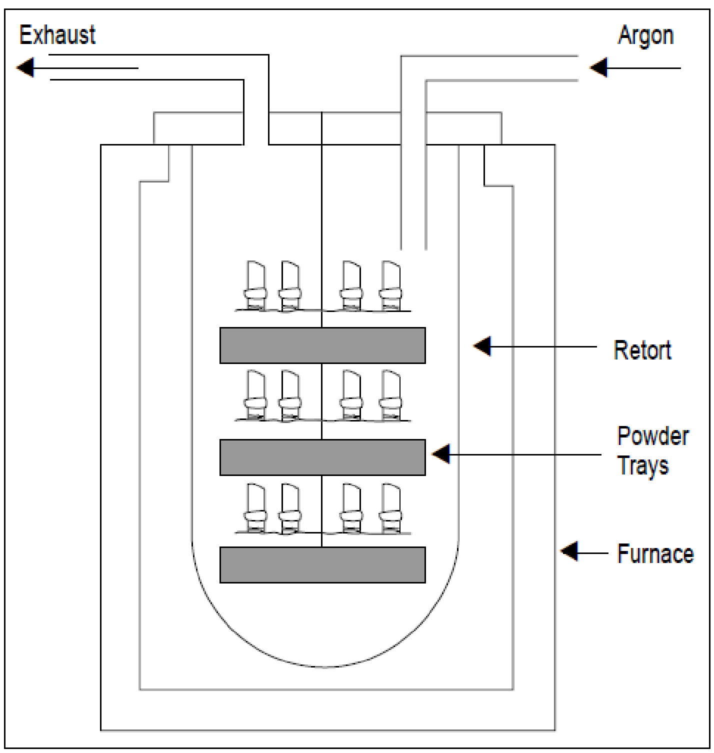



**Figure 13: A gas-phase CVD retort [18]**

### 2.4.2. Process parameters and coating morphology

Pack activity, process temperature, and time are three main process parameters for aluminizing to determine the coating morphology and thickness. Based on the pack activity, aluminizing can be classified as high-activity when aluminum diffuses inward (Figure 14) or low-activity when nickel diffuses outward (Figure 15).

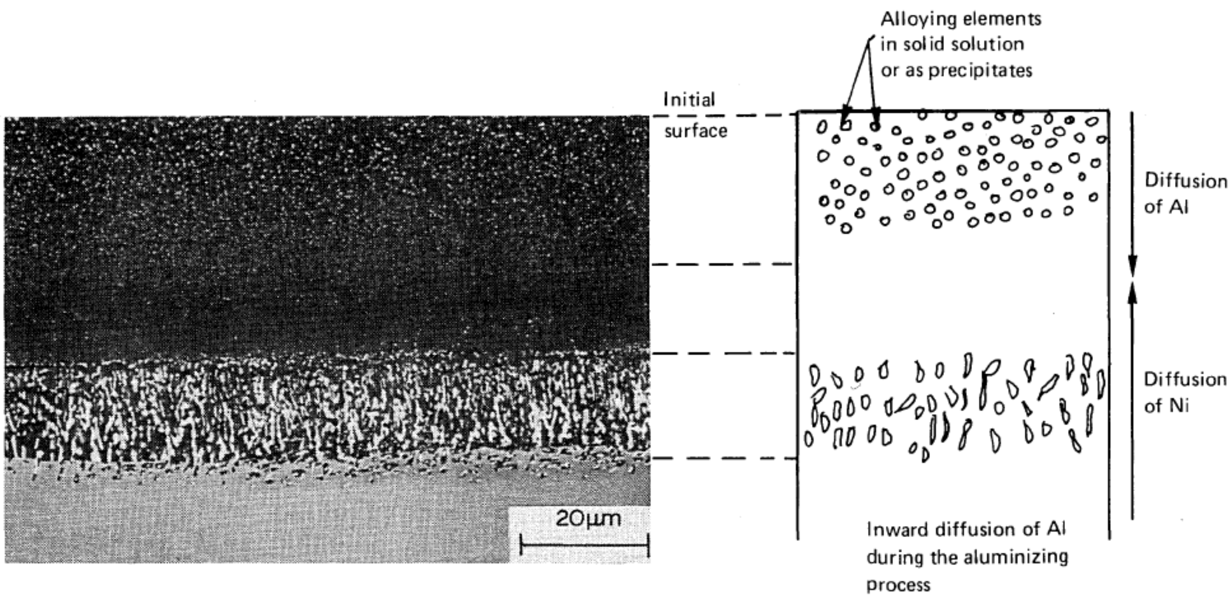



**Figure 14: High-activity pack cementation aluminide coating on CMSX-2; 7.5 h at 700°C in Cr-30Al pack, diffusion annealed at 1050°C for 16 h [19]**

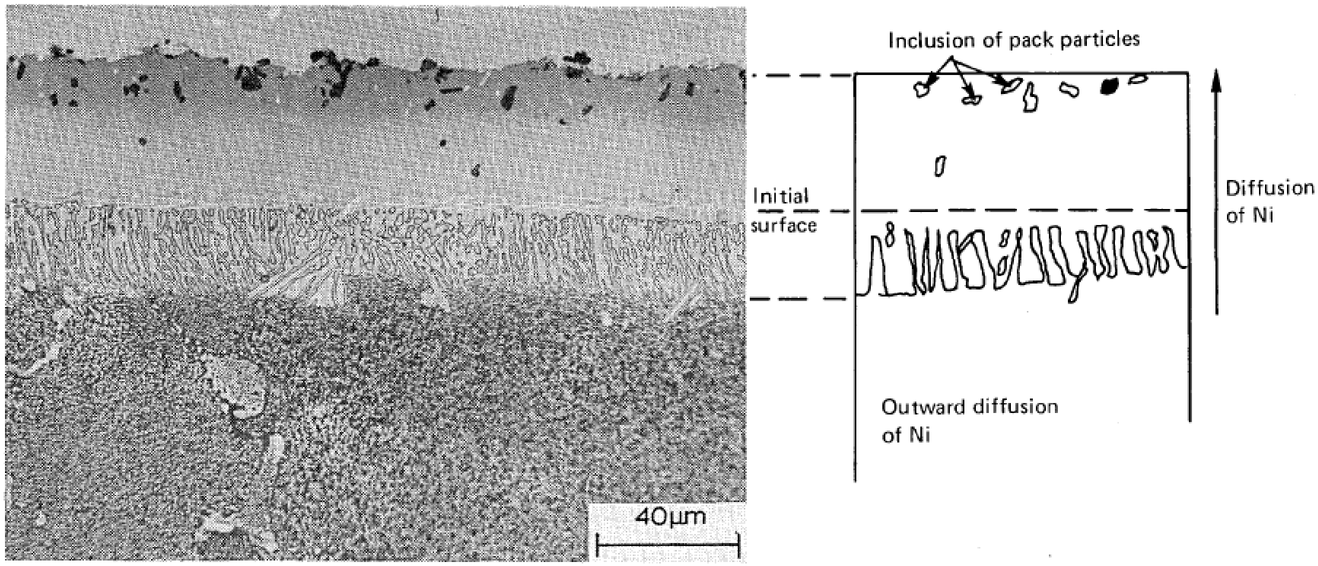



**Figure 15: Low – activity pack cementation aluminide coating on IN738 LC; 16 h at 1050°C with cement composed of 49 wt% (Cr-15Al), 1 wt% $NH_4Cl$, bal. $Al_2O_3$ [19]**

As Figure 14 shows, aluminum is the main diffusing species during high-activity aluminizing and the coating grows inward [19]. δ- $Ni_2Al_3$ forms at the substrate surface and it needs to be converted to a less brittle β-NiAl by annealing.

For low-activity aluminizing the coating grows beyond the initial surface due to the outward diffusion of nickel (Figure 15). β-NiAl forms at the surface and some pack particles may be entrapped in the coating.

Usually, the temperature for lower-activity aluminizing (about 1050°C) is higher than that for higher-activity aluminizing (about 700°C to 800°C). Therefore, a heat treatment after aluminizing may also be needed for substrate alloy properties recovery. The growth of diffusion coating thickness roughly obeys parabolic law, i.e. it is proportional to the square root of process time [19].

### 2.4.3. Phase equilibria in Ni-Cr-Al system by experiments

Inconel, is a series of nickel rich alloys where Cr is the dominant alloying element. The other alloying elements includes, Fe, Mo, Nb, Co, etc. Inconel alloys have good oxidation and corrosion resistance. Aluminizing is one approach to further increase the properties of inconel. The aluminide coating formed on the surface has very high hardness and stability and can be used in wear-resistant applications.

To understand the phase evolution during the aluminizing process, there is a lot of focus on phase equilibria for Ni-Cr-Al based alloys. Phase equilibria has been systematically investigated since the 1980s and were regularly updated. Ni-Cr-Al ternary isothermal sections at various temperatures were determined by the recent experimental investigation as shown in Figure 16.

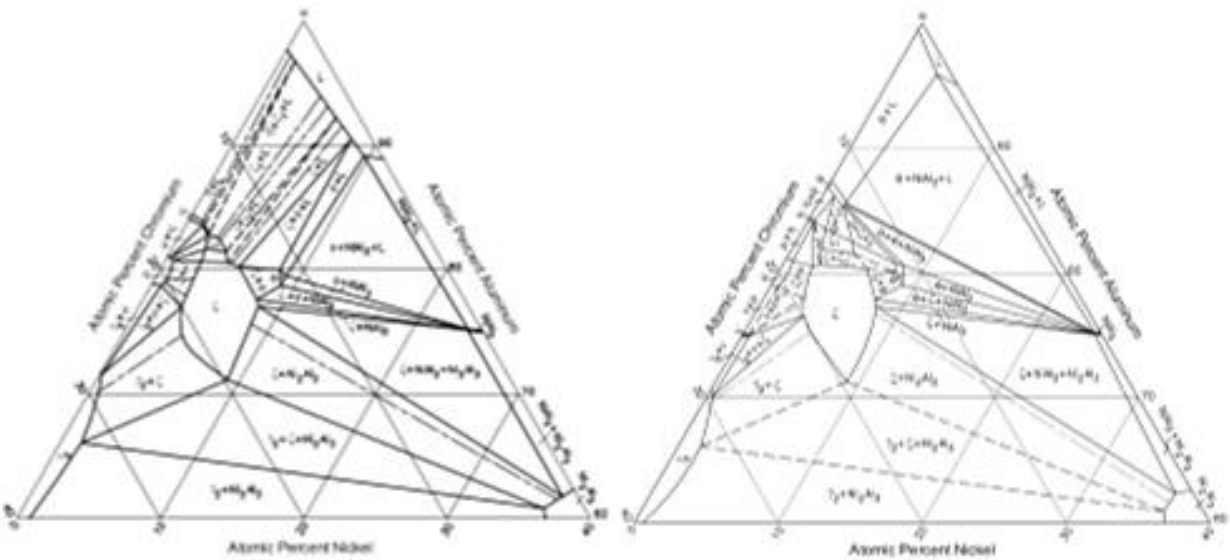


**Figure 16: Ni-Cr-Al Isothermal section at 800°C (left) and 900°C (right) determined by experimental investigation [20]**

### 2.4.4. Computational thermodynamics and its application in aluminizing

However, the shortcoming of the experimental investigation is that there is a lack of overall understanding of the system. Experiments need to the done again if the process condition is changed.

Due to this, computational thermodynamics have been developed from 1970s, known as the CALPHAD approach. CALPHAD is an acronym for the CALculation of PHAse Diagram. This program is capable of predicting the phase behavior in multi-component systems. In this approach, the Gibbs energy of individual phases is modeled and the model parameters are collected in a thermodynamic database.

Models for the Gibbs energy are based on the crystal structures of the phases. The development of CALPHAD techniques in the past three decades was discussed by Saunders and Miodownik [21]. The advantage of the CALPHAD approach is that once the accurate thermodynamic database is developed for the investigation system all the thermodynamics related properties can be predicted.

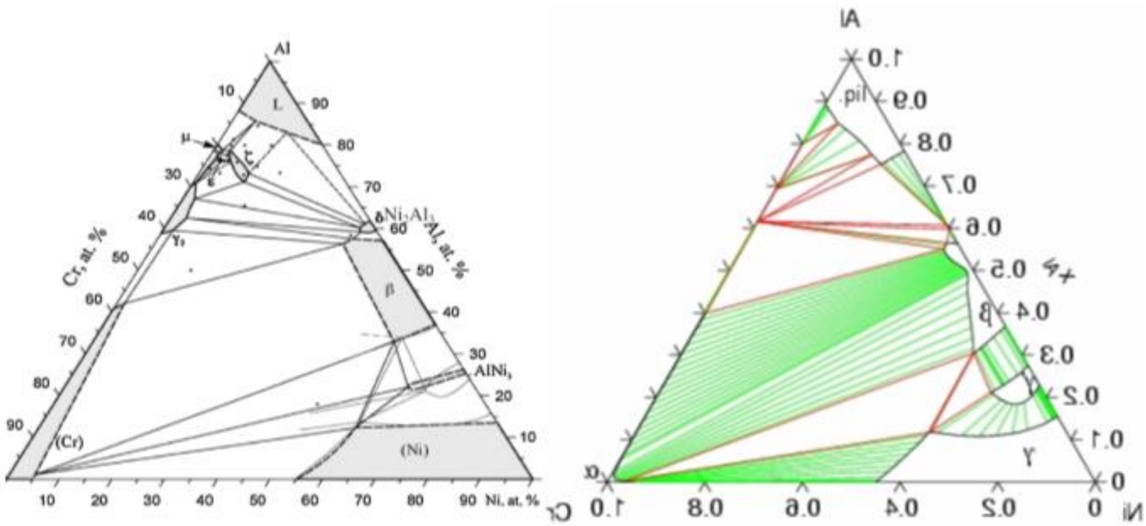



**Figure 17: Ni-Cr-Al Isothermal section at 1000°C (left) determined by experimental investigation [20] and calculated from TCNI5 (right)**

TCNI5 database can be used to make all the calculations for the Ni based alloys, which were carried out by using Thermo-Calc [22]. TCNI5 includes critically assessed thermodynamic descriptions for 23 elements and 292 phases. Most of the binary systems in this database have been assessed. TCNI5 also contains many assessed ternary systems, at least those being in equilibrium with γ and γ’ phase. The comparison between the calculated isothermal section of Ni-Cr-Al system at 1000°C was shown in Figure 17. It shows overall pretty good agreement with the experimental investigation.

# 3.0 Methodology and results

## 3.1. Survey results and analysis

A survey, seen in Appendix A-Blank Survey, was filled out by numerous attendees at the Center for Heat Treating Excellence meeting held at Worcester Polytechnic Institute in December of 2012. The purpose of gathering this information from professionals in the heat treating industry was to obtain a more realistic grasp on what alloys need life improving, what parts typically fail, and why these industry professionals think their parts are failing. After reading through the surveys it was hoped that the project could move forward with more purpose and direction given that the problem statement is so broad considering all the different types of furnace parts and fixtures, what they are made of, and the various types of atmospheres these materials encounter. The survey results were compiled into one document that can be seen in Appendix B-Survey Results.

Table 6 shows important properties necessary for various heat resistant alloys that either industry professionals said were being used in their company's furnaces or were mentioned in literature. Further details of each of these alloys including mechanical, thermal, and physical properties, chemical composition of the alloy, what parts the alloy should be used for, and what it should not be used for will eventually be written out and compiled into a catalog for the CHTE and industry use. Given this booklet it will be easier for one to decide the proper alloy for use in a particular situation and to first consider the failure modes of alloys before using them in a specified application.

Table 6: Properties of heat resistant materials for use in high temperature applications

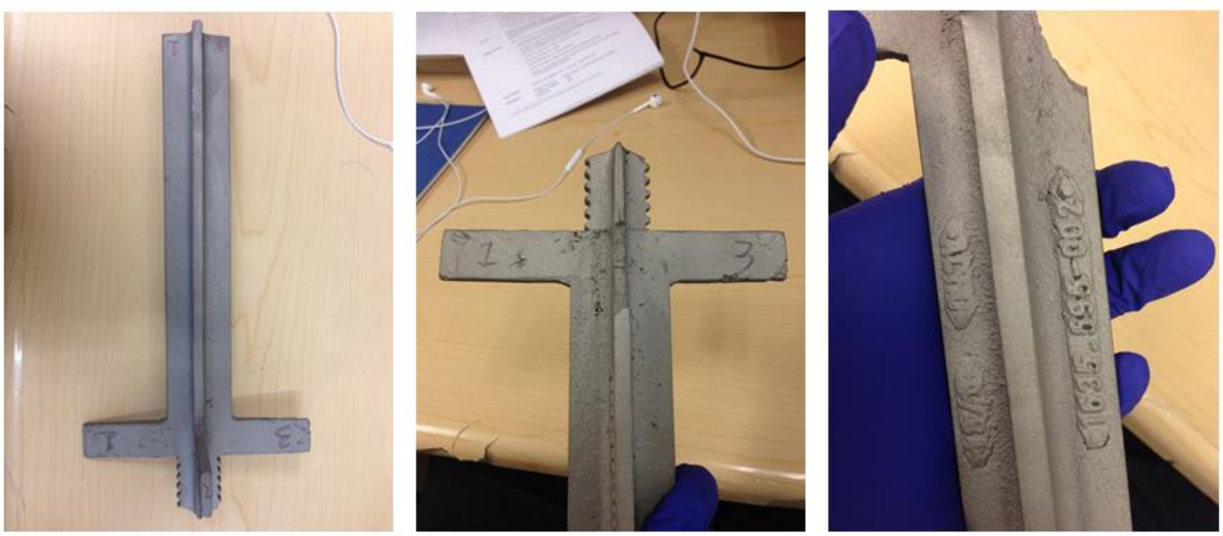
## Heat Resistant materials

| Materials | Density | Melting Point | Thermal Conductivity | Oxidation Resistance | Heat Capacity | Thermal Expansion | Modulus of Elasticity |
|---|---|---|---|---|---|---|---|
| Unit | lb/in$^3$ | °F | Btu*ft/ft$^2$*hr*°F (70°F) | °F | Blu/lb*°F | $10^{-6}$/°F @1000°F | $10^6$psi |
| RA304 | 0.289 | 2650 | 9.4 | 2200 | 0.12 | 10 | 29 |
| RA333 | 0.294 | 2375-2450 | 6.4 | | 0.072 | 8.6 | 29.2 |
| 304L | 0.285 | 2550-2590 | 9.4 | | 0.12 | 9.2 | 29 |
| 330 | 0.292 | 2450-2540 | 7.2 | 2000 | 0.11 | 9.7 | 29 |
| 314 | 0.215 | | 10.1 | | 0.12 | 8.3 | 29 |
| Incoloy 800 | 0.287 | 2475-2525 | 6.67 | | 0.11 | 9.4 | 28.5 |
| Inconel 600 | 0.304 | 2498-2576 | 8.59 | | 0.106 | 8.1 | 30 |
| Inconel 601 | 0.293 | 2375-2495 | 11.6 | 2200 | 0.107 | 8.5 | 29.95 |
| Inconel 617 | 0.302 | 2430-2510 | 12.4 | | 0.1 | 7.7 | 30.6 |
| 800H | 0.287 | 2475-2525 | 6.7 | | 0.11 | 9.9 | 28.5 |
| 214 | 0.291 | 2475-2550 | 6.92 | | 0.108 | 8.2 | 31.6 |
| 230 | 0.324 | | 5.16 | | 0.095 | | |
| HT | 0.286 | 2450 | 7 @212°F | | 0.11 | 9.6 | 27 |
| HK | 0.28 | 2540 | 7.9 @212°F | | 0.12 | 9.8 | 20-27 |
| HN | 0.283 | 2500 | 7.5 @212°F | | 0.11 | 9.7 | 27 |
| HU | 0.29 | 2510 | 7 @212°F | | 0.11 | 8.8 | 27 |
| HX | 0.297 | 2300-2470 | 81 Btu/fth°F | | 0.11 | 8.27 | 25 |
| RA253MA | 0.282 | 2500-2610 | 8.6 | 2000 | | 14.3@1400F | 29 |
| RA602CA | 0.285 | 2350-2550 | 11.6@1000F | 2200 | | 8.2 | 30 |
| Hastelloy X | 0.297 | 2300-2470 | 63 | | 0.116 | 8.1 | 29.8 |

## 3.2. Failure analysis of furnace fixture

It was necessary to obtain a failed part from a furnace to infer about what was happening in some of these furnace parts and fixtures to be able to compare with literature and to make new hypotheses.

Members of the Center for Heat Treating Excellence, Company E, provided a broken racking post fixture of unknown material for the group to analyze. The material was said to most likely be HT, however, this needs to be justified and made sure of. Figure 18 below shows three different views of the racking post. The post was in both vacuum and endothermic carburizing furnaces at a loosely approximated time of 30 hours per week for 5 to 10 years. The vacuum carburizing operates at 1700-1900°F and the endothermic carburizing furnace ran from 1500-1700°F.



(a) (b) (c)

**Figure 18: Failed unknown racking post sample from Company E's furnaces. The post was labeled for reference purposes. (a) Normal view of the entire part (b) front view of broken piece (c) close-up side view of broken section.**

Figure 19: Sample cut, mounted, and polished for testing

The first sample was cut from the bottom of the racking post and was used to determine the unknown elements in the material and to take an initial look at the part. Figure 19 shows the sample that was cut from the bottom of the racking post, mounted, and then polished for testing purposes. Optical microscope pictures were first taken and observed. These pictures were taken from the outward most surface of the part to the interior to observe changes in the parts appearance (Figure 21).

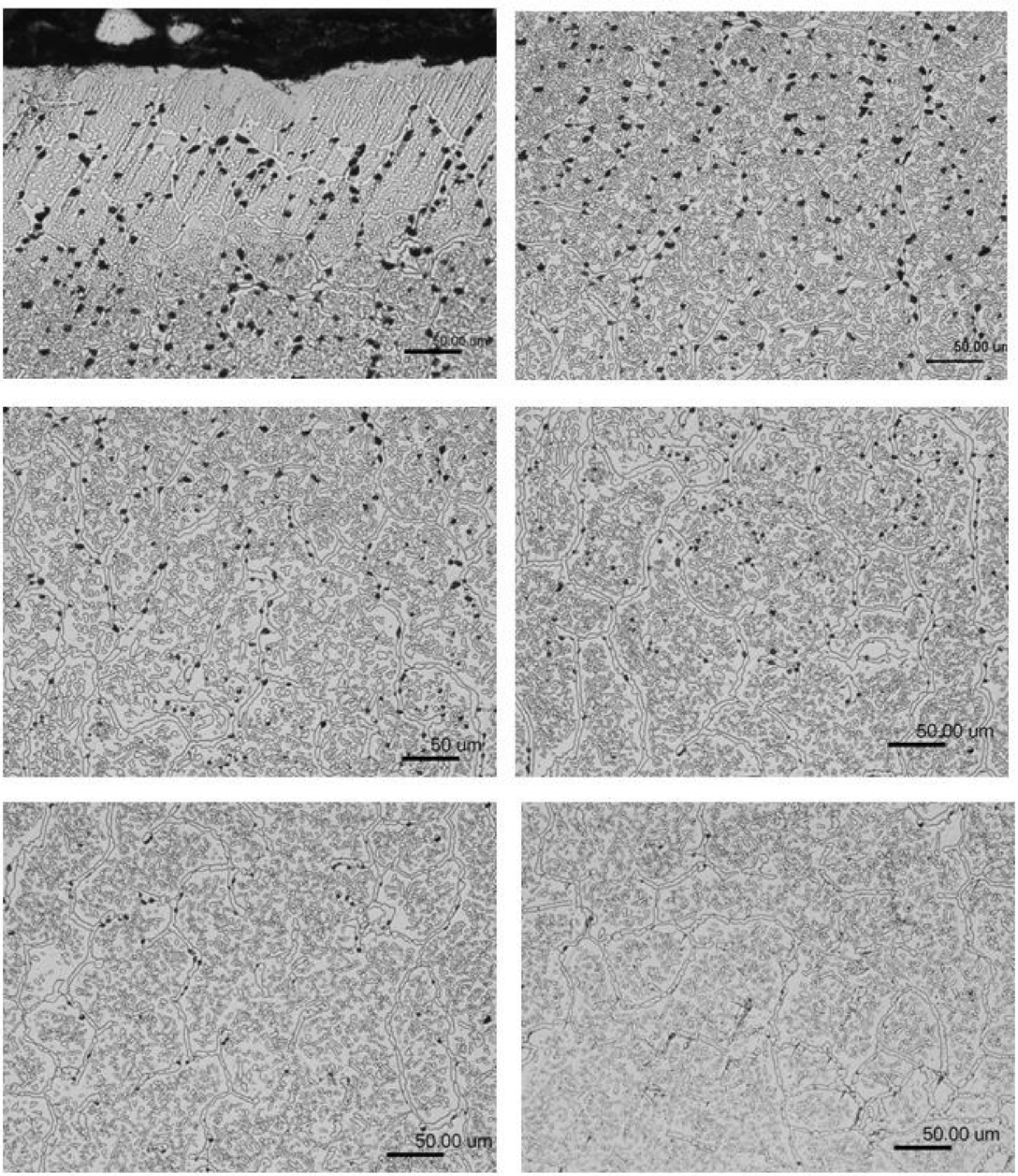



**Figure 20: Optical microscope pictures of sample. Outermost edge (top left) to core (bottom right) of the sample (50µm)**

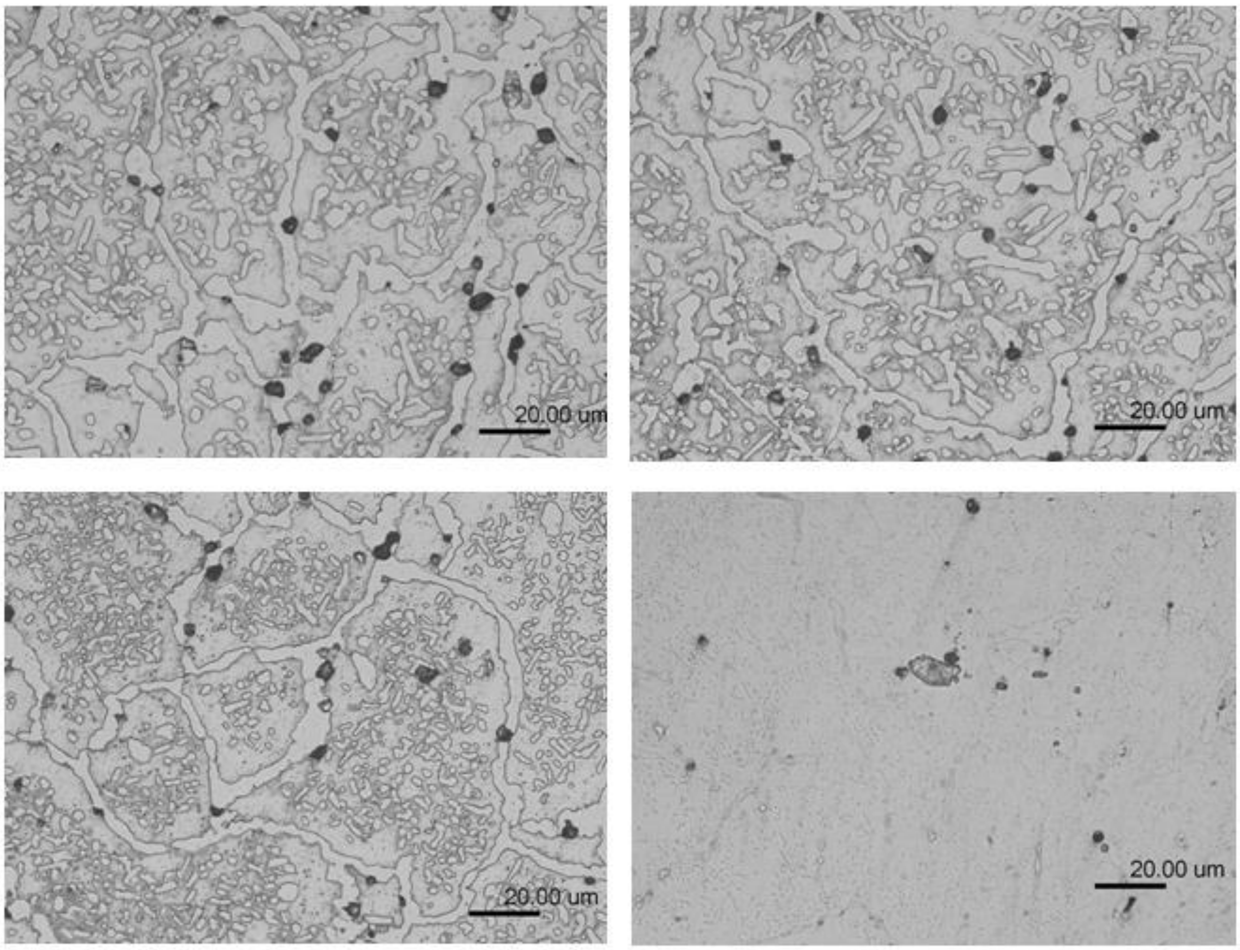



**Figure 21: Optical microscope pictures of sample. Outermost edge (top left) to core (bottom right) of the sample (20µm)**

Cracking is evident toward the outer surface of the part, as well as possible carburization defects (Figure 21). These defects become fewer and further between as the interior of the part is reached. Other pores can be seen in the interior of the part that may be a result of casting. It is imperative to study this gradient and decide if there is more or less of an element as one moves through the diffusion zone.

Electron dispersion X-ray spectroscopy (EDS) and Optical Emissions Spectroscopy (OES) techniques were both utilized to obtain the chemical composition of the unknown sample such that it can be compared with standard heat resistant alloys to figure out what material the racking post is made of. Once the material is determined failure analysis justifications can be made.

The EDS results, found in Appendix C-EDS Results, showed significant amounts of elemental C, Cr, Fe, Ni, O, and Al as well as small amounts of Si, P, S, Cu, and Mn. However, there are some constraints with EDS and the accuracy of X-ray spectroscopy is questionable so it must be compared with the more accurate OES results (Appendix D-OES Results).

Table 7 below shows the average percent compositions of each element; approximately 44% Fe, 34% Ni, and 17% Cr was found. The carbon concentrations found in both the tests varied too much to determine the exact amount of carbon in the sample. This variation is most probably due to carburization of the part, as it spent many hours in a heat treating furnace. High concentrations of iron, nickel, and chromium agree with both tests as well. The aluminum oxides found in the EDS results is most probably due to the preparation of the sample. As during this preparation aluminum is introduced. Other trace elements were found in both the OES and EDS reports.

**Table 7: Optical Emissions Spectroscopy result summary of unknown racking post material, Appendix D-OES Results.**

| Optical Emissions Spectroscopy (OES) Results | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Unknown Racking Post | | | | | | | | | | | | | |
| Element | Fe | Ni | Cr | Mo | Co | Si | W | P | S | C | Mn | Nb | Cu |
| | % | % | % | % | % | % | % | % | % | % | % | % | % |
| 1 | 45.61 | 32.76 | 17.69 | 0.29 | 0.21 | 1.88 | 0.12 | 0.04 | 0.03 | 0.27 | 0.35 | 0.19 | 0.15 |
| 2 | 44.14 | 33.83 | 16.53 | 0.29 | 0.21 | 1.99 | 0.11 | 0.03 | 0.02 | 1.86 | 0.32 | 0.20 | 0.15 |
| 3 | 44.23 | 33.62 | 16.67 | 0.29 | 0.21 | 1.98 | 0.11 | 0.03 | 0.02 | 1.84 | 0.32 | 0.20 | 0.15 |
| 4 | 43.68 | 33.91 | 16.28 | 0.29 | 0.21 | 2.03 | 0.11 | 0.04 | 0.01 | 2.47 | 0.30 | 0.18 | 0.25 |
| 5 | 43.73 | 33.88 | 16.20 | 0.29 | 0.21 | 1.99 | 0.10 | 0.03 | 0.01 | 2.58 | 0.30 | 0.18 | 0.25 |
| 7 | 45.58 | 32.55 | 17.86 | 0.30 | 0.22 | 1.92 | 0.12 | 0.04 | 0.03 | 0.29 | 0.36 | 0.19 | 0.16 |
| 8 | 42.27 | 35.28 | 16.82 | 0.23 | 0.20 | 2.07 | 0.09 | 0.02 | 0.02 | 2.11 | 0.28 | 0.22 | 0.28 |
| 9 | 43.11 | 34.93 | 17.17 | 0.22 | 0.20 | 2.04 | 0.09 | 0.03 | 0.02 | 1.27 | 0.30 | 0.21 | 0.17 |
| 10 | 43.05 | 34.96 | 17.07 | 0.22 | 0.20 | 2.05 | 0.09 | 0.03 | 0.02 | 1.41 | 0.29 | 0.21 | 0.17 |
| 11 | 44.61 | 33.89 | 17.84 | 0.22 | 0.21 | 1.90 | 0.10 | 0.02 | 0.02 | 0.26 | 0.31 | 0.19 | 0.17 |
| 12 | 44.20 | 34.06 | 17.96 | 0.23 | 0.21 | 1.98 | 0.10 | 0.03 | 0.02 | 0.28 | 0.32 | 0.20 | 0.17 |
| **Avg** | **44.02** | **33.97** | **17.10** | **0.26** | **0.21** | **1.98** | **0.10** | **0.03** | **0.02** | **-** | **-** | **-** | **-** |

Research was completed to compare the results of the unknown sample to that of known heat resistant materials.

Table 8 below shows the known chemical compositions of HT alloy. After looking at the two results and many other chemical compositions of various cast and wrought alloys it is safe to infer that the racking post was made out of HT cast alloy. Other similar alloys such as wrought 800H have less silicon and carbon concentrations than that of HT and less than what was seen in these results. Although there are low levels of Si in the sample, Si is crucial in cast alloys as it is a component that makes the alloy castable which helps draw these conclusions as well.

Table 8: Actual chemical composition of HT alloy [23, 24]

| HT Alloy | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | C | Mn | Si | Cr | Ni | Mo | P | S |
| Min % | 0.4 | 0.35 | 0.35 | 15 | 33 | - | - | - |
| Max % | 0.5 | 2 | 2 | 19 | 37 | 0.5 | 0.04 | 0.04 |

These results can then be compared to the known microstructure of HT to determine the failure mode of HT alloy when it is used in high temperature applications. HT has good resistance to thermal shocks, oxidation, and carburization at high temperatures, however is vulnerable in high-sulfur gases [2]. As mentioned in the literature review HT is used in load-bearing situations such as for radiant tubes, retorts, and in this particular case for fixtures[2]. In HT nickel strengthens the matrix of the material and promotes austenitic growth, stabilizing the face-center cubic austenite phase [2]. Both chromium and nickel promote resistance to oxidation, whereas chromium improves creep properties and rupture strengths[2]. Figure 22 shows the microstructure of a cast HT-44 alloy from volume 9 of the ASM Handbook: Metallography and Microstructures. HT is comprised of an austenitic face-center-cubic matrix with eutectic carbides on the boundaries [25]. The carbides on the grain-boundaries of HT help prevent the grains from sliding, increase hardness, and allow for stress relaxation to occur [25].

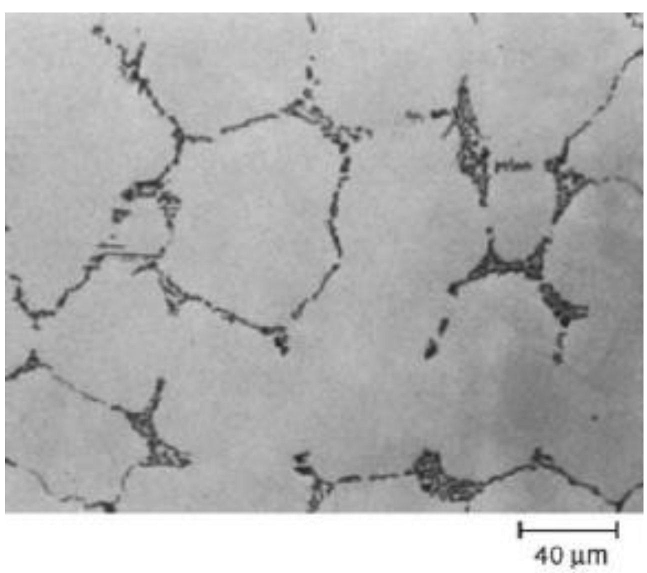



Figure 22: Cast HT-44 microstructure [25].

In comparing Figure 22 with the multiple pictures in Figure 20 it is evident that the picture of HT-44 compares most prominently with the core of the HT sample. Also by observing a view of the sample at a lesser magnification in Figure 23, one can conclude that there is a significant gradient over 1mm of the part. Given Figure 19 the center of the optical microscope pictures in Figure 23 should be of a lighter, more shinny color than they appear. The outer 1mm gradient of the sample part is actually of a more dull appearance; however it is still clear that there is a difference in material across the part.

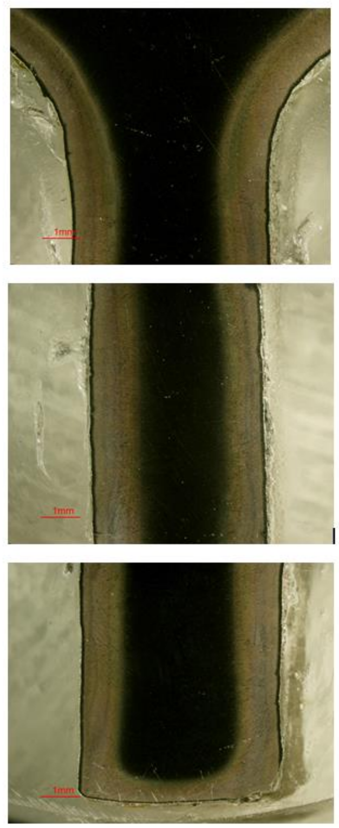



**Figure 23: Diffusion zone; 1mm**

The gradient shown in Figure 23 is most likely a layer due to an excess in carbon diffused into the part. This conclusion can be confirmed by a microhardness profile. Carbon concentrations in the sample part relate directly to the hardness of the overall alloy. The higher the carbon concentration the greater the hardness of the material. Figure 24 below shows a significant, constant, and consistent decrease in the hardness value from the outside of the test HT sample to the inner depths of the sample.

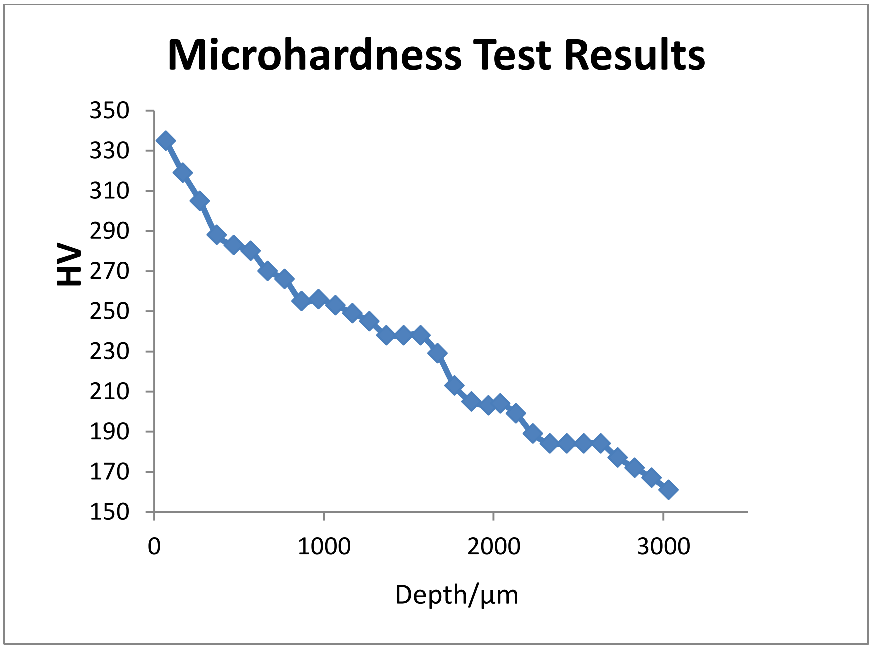



Figure 24: Micro-hardness test results

## 4.0 Conclusions

Given survey results obtained from members of CHTE it is evident that there are still many materials used in the heat treatment industry that need to be investigated. Test methods to measure the performance of an alloy in gas carburizing, high temperature vacuum (low pressure) carburizing, and gas carbonitriding furnaces needs to be investigated. Ideally a standardized method to compare the effects of thermal fatigue, and or carburization attack on various alloys should be obtained as well. Surface treatments, both silicon-rich and aluminum-rich, should be tested on a variety of alloys (if possible), heat treated for a substantial amount of time, and compared to their uncoated counterparts. For example, aluminizing a part could enhance corrosion resistance, carburization resistance, and creep.

Although HT has good carburization resistance it is still evident that this particular HT sample failed due to excess carbon content in the matrix. Since it is not known exactly how long the HT racking post was in the furnace for, what type of furnace(s) the sample has seen, or how the sample was cycled it is impossible to draw a concrete conclusion as to whether or not the racking post, although failed, performed a relatively acceptable life in industry.

The breadth of this problem opens doors to a variety of other options to consider investigating. Redesign of fixtures could be considered to improve their life. Quenching fixtures multiple times results in significant thermal fatigue, uniformly quenching these parts would help reduce fatigue. Fixtures made of a lower weight and that have a low heat capacity should be considered for redesign. Also, reducing high stress areas in fixtures by redesign may be advantageous. Furthermore, parts in furnaces that see significant damage due to metal dusting are those that reside somewhere in the furnace where gaseous flows are stagnant. Somehow reducing these flows by possibly changing the design of furnaces may reduce failures.

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## 6.0 Appendix A-Blank Survey

**Survey for CHTE Alloy Life Improvement project** – Dec 05, 2012

(Improvements to life of alloy in heat treatment furnaces and part holding fixtures)

Name: ________________________ Company: ________________________

Date: _________________________

This survey will be used in the CHTE project, on improvements to life of alloy in furnaces and fixtures.

A) ***What are your biggest problems with heat treatment furnace alloys and part holding fixtures?***

B) ***What alloys are you using for 1. furnace parts and 2. fixtures used in heat treatment processes?***

1. Furnace parts

2. Fixtures

***C) <u>Who are the suppliers of these parts?</u>***

***D) <u>How often do these fixtures or furnace parts need replacement?</u>***

***E) <u>What do you believe to be the most common failure mode of these alloys?</u>***

F) ***What test methods might be used to measure the alloys performance in the furnace or fixture.***

G) ***Any other suggestion?***

## 7.0 Appendix B-Survey Results

# Survey: CHTE Alloy Life Improvement Project

A) What are your biggest problems with heat treatment furnace alloys and part holding fixtures?

***Company A:***

- Bending with alloy fixtures.
- Cost of C/C fixture.
- Avoiding contact reaction between C/C and the products at temperature > 1050℃

***Company B:***

- Distortion of fixtures, which then prevent us from holding parts in the proper orientation.
- Cracking at welds of non-cast baskets.

***Company C:***

- Cracks/broken corners on cast grids/ trays.
- Warpage of baskets and hanging bars.
- Warpage of vertical posts.
- Warpage of grids.
- Failure/breaking of mesh belts.
- Holes in radiant tubes.

***Company D:***

- Life time of continuous furnace wire mesh belts (314S/S typical standard belt metal)
- PM Sintering at 2150°F in 10%$H_2$/90%$N_2$ atmospheres

**Company F*:***

- Distortion of fixtures.

***Company E:***

- Sooting
- Distortion

B) What alloys are you using for 1. Furnace parts and 2. Fixtures used in heat treatment processes?

I. Furnace parts

***Company A:***

- Graphite
- C/C
- Molybdenum

***Company E:***

- HT
- Molybdenum

***Company D:***

- 314S/S Belts

***Company C:***

- Radiant tubes (Most expensive, frequent coats)
- Rollers in roller hearth furnaces
- Rollers in IQ furnaces (Integral Quench, designed for processes such as annealing, carburizing, clean hardening and carbonitriding.
- Fan failures
- 304 bars for hanging parts on
- 330 bars for hanging parts on
- 253 MA bars for hanging parts on (253 MA bar, high strength heat resistant stainless steel)
- Hu cast grids base trays (Sometimes HX, HT)
- 330 welded baskets
- Inconel welded baskets
- Mesh belts
- Hu posts, crossbeams.

***Company B:***

- M-2 (high speed tool steel), A-2 (Tool steel), H13, 52100, 4140

II. Fixtures

***Company B:***

- Skids & Trays: Molybdenum

- Baskets: Inconel 600 --From Wireco
  PTS220, PTS236 --From Protech

  Wire grades: Hu, HT

***Company D:***

- Muffles- 330S/S

***Company F:***

- RA333
- Inconel 600

***Company E:***

- HT
- Mancellium

***Company A***

- High Nickel Alloy
- C/C

C) Suppliers

***Company A:***

1. Alloy:
   - NAG
   - Castalloy
   - Alcon
2. C/C:
   - SGC
   - Graphite Materials

***Company B:***

- Wirco
- Protech

***Company F:***

- Roller alloy
- Steel-tech

***Company E:***

- Wirco
- Cronite

***Company D:***

- Maryland Wire Belt among others
- Sinterite and Abbott (Furnace manufacturers) - Continuous Belts furnace manufactures purchase belts from various vendors

  Powder metal parts manufacturers:

  - GKN
  - Metaldyne
  - MPP

D) Lifetime

***Company A:***

- Alloy fixtures: 2-3 years
- C/C: > 10 years

***Company B:***

- 1-3 years (Three years is ideal situation. We have had some poorly handled baskets that have not lasted long at all.)

***Company E:***

- HT: 5 years
- Mancellium: Still new.

***Company D:***

- 9-12 months (Depending primarily on Belt/Weight part loading.)

E) Most common failure mode of these alloy

- Thermal fatigue of alloy fixtures
- Cracking, Distortion.
- Operation handling
- Water quenching
- Rough handling
- Carburizing
- Lowering eutectic/fatigue/overloading/Thermal stress

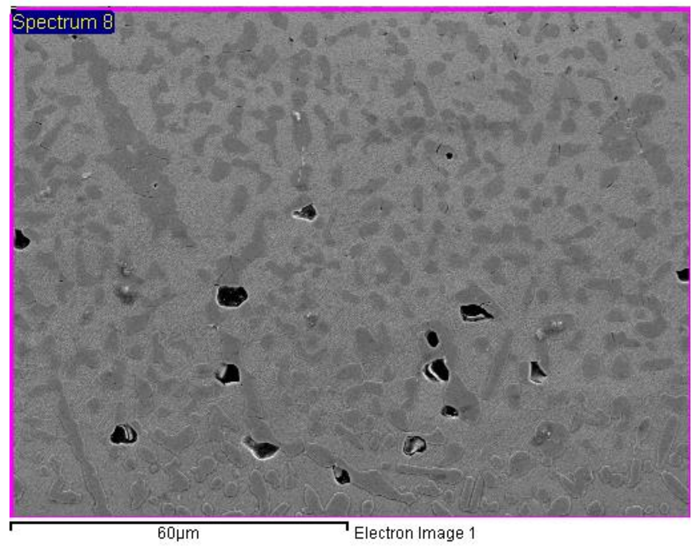
F) Test that used to measure the alloys performance in the furnace or fixture.

- Extension of belt life/fatigue strength
- Field test under production conditions
- Thermal cycling
- Mechanical abuse
- Dimensional stability

G) Suggestion

- Change in alloys for belt, ex. 314s/s vs Inconel 601
- Coatings-using ceramic $Al_2O_3$ and Nitrides/ Borides
- C/C coated with ceramic coating has been tested in the past without success yet (Has been published in Germany)

# 8.0 Appendix C-EDS Results




Spectrum processing :

No peaks omitted

Processing option : All elements analyzed (Normalised)

Number of iterations = 3

Standard :

C CaCO3 1-Jun-1999 12:00 AM

Si SiO2 1-Jun-1999 12:00 AM

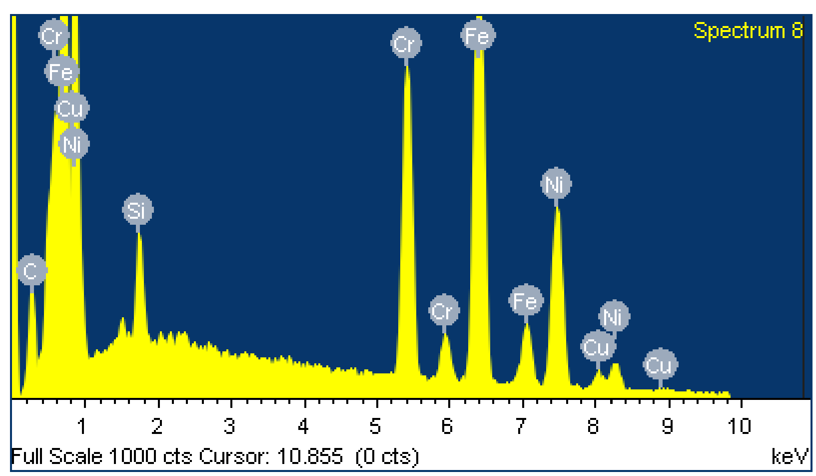
Cr Cr 1-Jun-1999 12:00 AM

Fe Fe 1-Jun-1999 12:00 AM

Ni Ni 1-Jun-1999 12:00 AM

Cu Cu 1-Jun-1999 12:00 AM

| Element | Weight% | Atomic% |
|---|---|---|
| C K | 7.22 | 26.40 |
| Si K | 1.67 | 2.61 |
| Cr K | 17.09 | 14.43 |



# Project 1

4/2/2013 2:30:14 PM

Spectrum processing :

Peaks possibly omitted : 2.160, 2.625 keV

Processing option : All elements analyzed (Normalised)

Number of iterations = 4

Standard :

C CaCO3 1-Jun-1999 12:00 AM

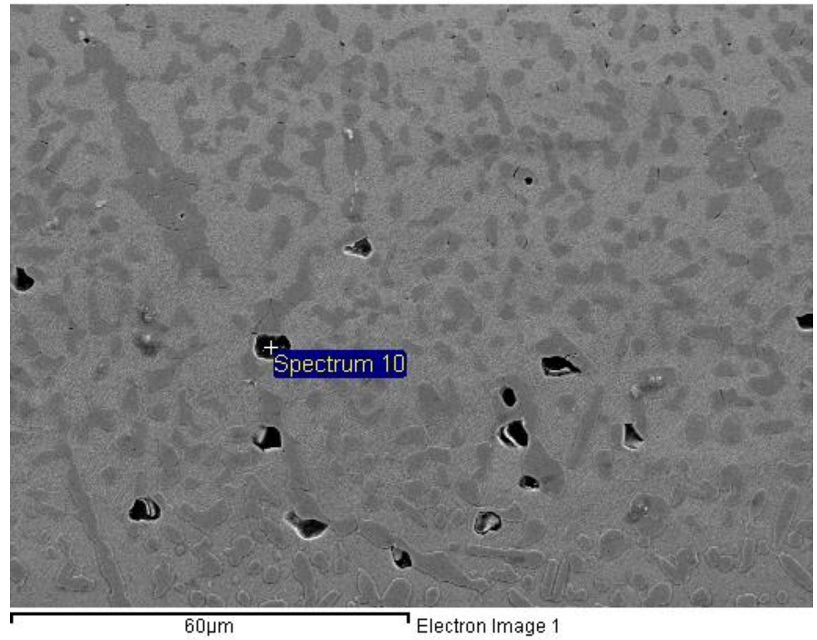
O SiO2 1-Jun-1999 12:00 AM

Al Al2O3 1-Jun-1999 12:00 AM

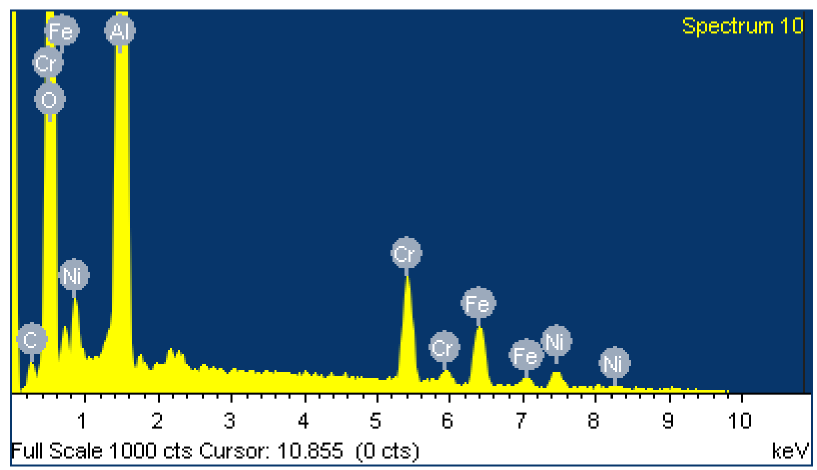
Cr Cr 1-Jun-1999 12:00 AM

Fe Fe 1-Jun-1999 12:00 AM

Ni Ni 1-Jun-1999 12:00 AM




| Element | Weight% | Atomic% |
|---|---|---|
| C K | 2.47 | 4.46 |
| O K | 43.10 | 58.37 |
| Al K | 38.36 | 30.80 |




Comment:

# Project 1



Spectrum processing :

No peaks omitted

Processing option : All elements analyzed (Normalised)

Number of iterations = 3

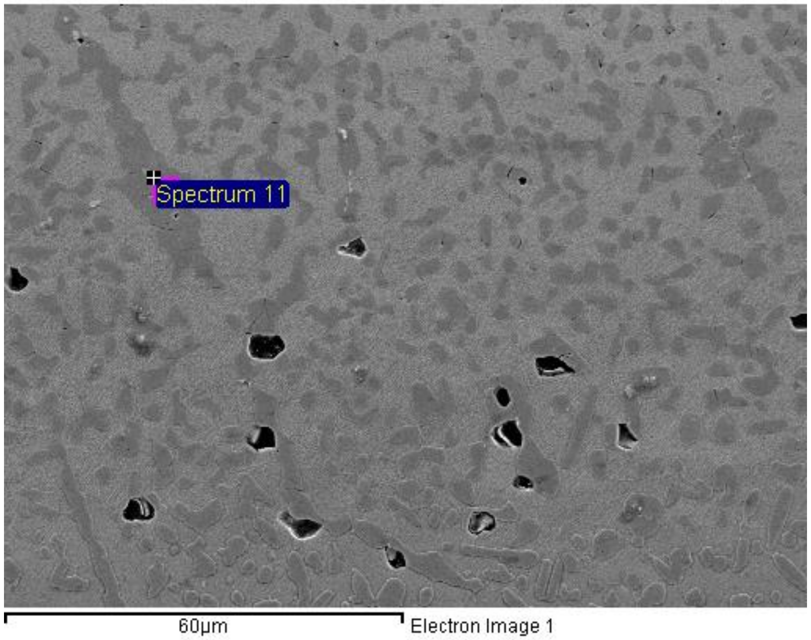
Standard :

C CaCO3 1-Jun-1999 12:00 AM

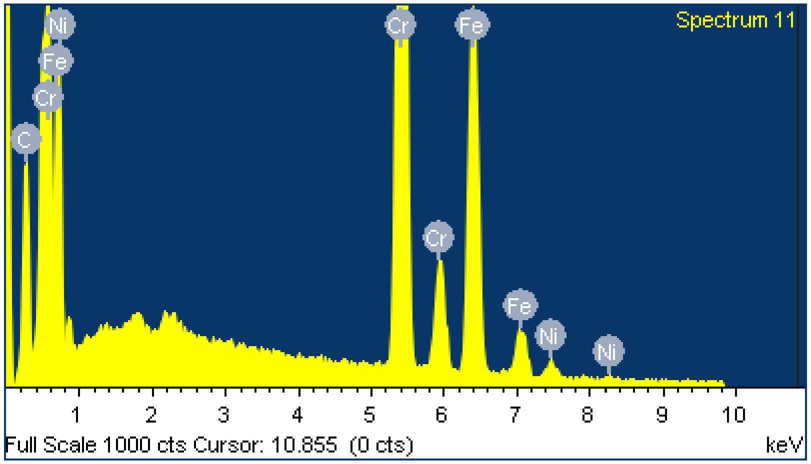
Cr Cr 1-Jun-1999 12:00 AM

Fe Fe 1-Jun-1999 12:00 AM

Ni Ni 1-Jun-1999 12:00 AM




| Element | Weight% | Atomic% |
|---|---|---|
| C K | 12.90 | 39.86 |
| Cr K | 47.90 | 34.18 |
| Fe K | 36.77 | 24.43 |
| Ni K | 2.43 | 1.54 |




Comment:

Spectrum processing :

No peaks omitted

Processing option : All elements analyzed (Normalised)

Number of iterations = 3

Standard :

C CaCO3 1-Jun-1999 12:00 AM

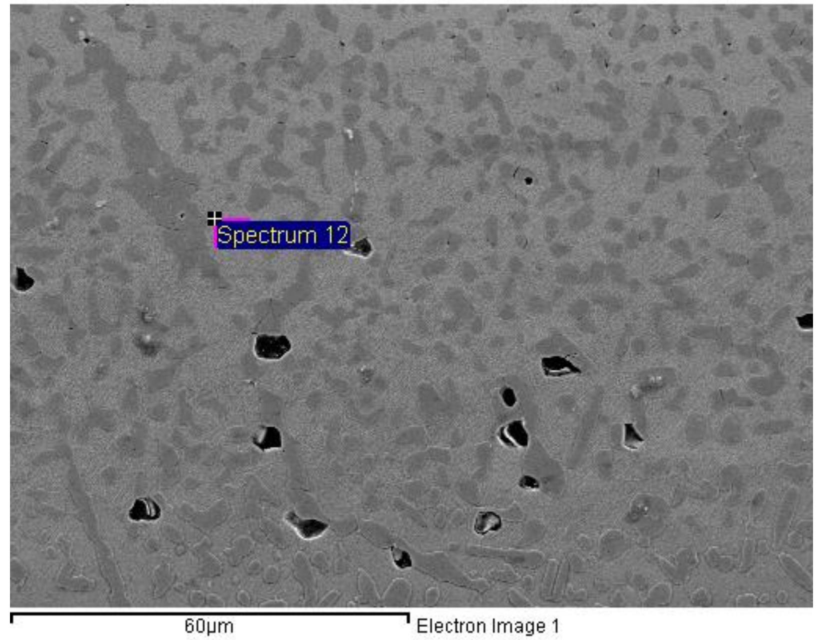
Si SiO2 1-Jun-1999 12:00 AM

Cr Cr 1-Jun-1999 12:00 AM

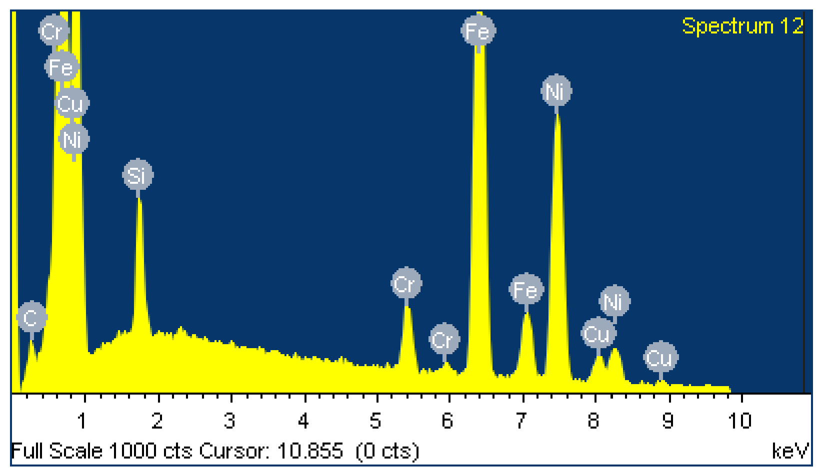
Fe Fe 1-Jun-1999 12:00 AM

Ni Ni 1-Jun-1999 12:00 AM

Cu Cu 1-Jun-1999 12:00 AM




| Element | Weight% | Atomic% |
|---|---|---|
| C K | 2.26 | 9.78 |
| Si K | 2.22 | 4.10 |
| Cr K | 3.33 | 3.32 |




Comment:

# Project 1

4/2/2013 2:35:14 PM

Spectrum processing :

No peaks omitted

Processing option : All elements analyzed (Normalised)

Number of iterations = 4

Standard :

C CaCO3 1-Jun-1999 12:00 AM

O SiO2 1-Jun-1999 12:00 AM

Al Al2O3 1-Jun-1999 12:00 AM

Si SiO2 1-Jun-1999 12:00 AM

P GaP 1-Jun-1999 12:00 AM

S FeS2 1-Jun-1999 12:00 AM

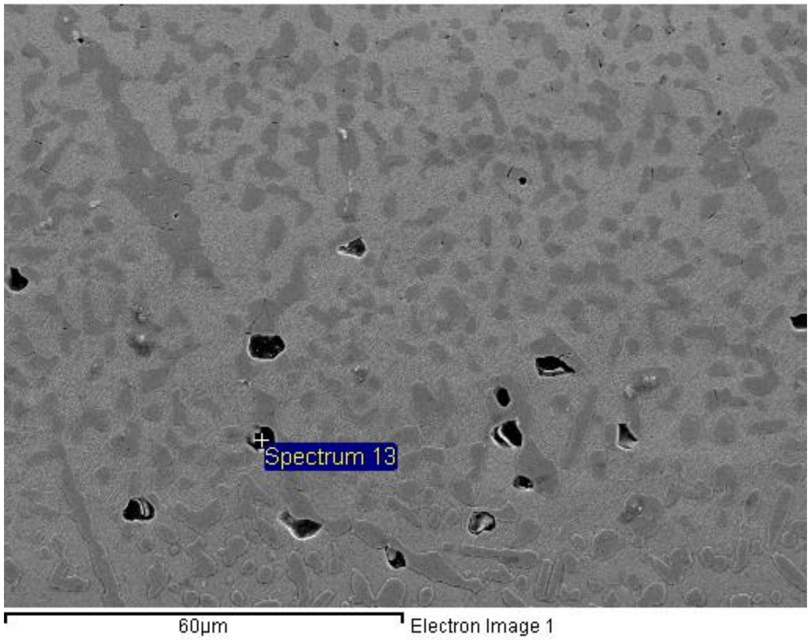
Cr Cr 1-Jun-1999 12:00 AM

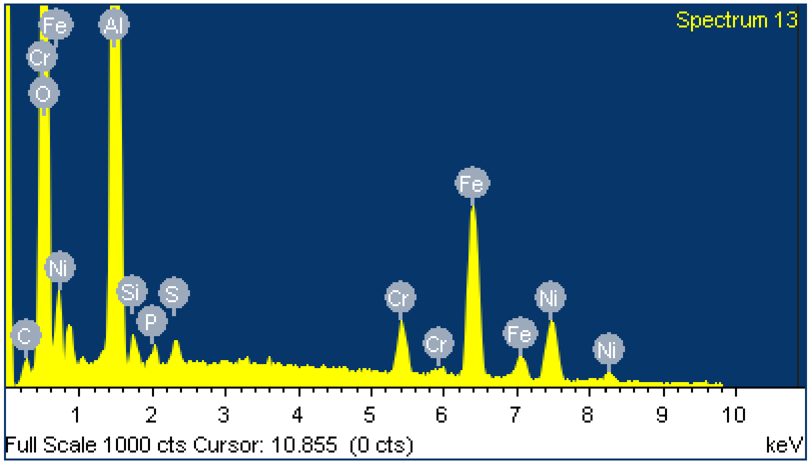
Fe Fe 1-Jun-1999 12:00 AM

Ni Ni 1-Jun-1999 12:00 AM

| Element | Weight% | Atomic% |
|---|---|---|
| C K | 2.21 | 4.53 |

Comment:



# Project 1

4/2/2013 2:38:12 PM

Spectrum processing :

Peak possibly omitted : 2.000 keV

Processing option : All elements analyzed (Normalised)

Number of iterations = 4

Standard :

C CaCO3 1-Jun-1999 12:00 AM

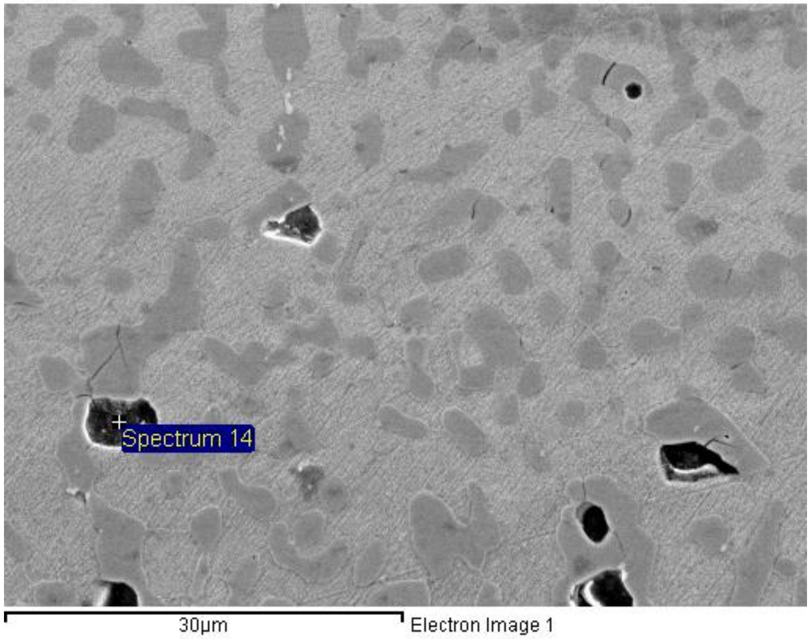
O SiO2 1-Jun-1999 12:00 AM

Al Al2O3 1-Jun-1999 12:00 AM

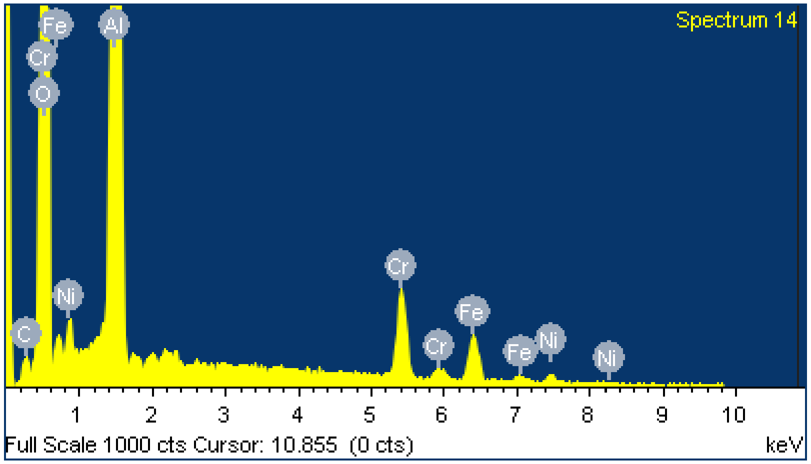
Cr Cr 1-Jun-1999 12:00 AM

Fe Fe 1-Jun-1999 12:00 AM

Ni Ni 1-Jun-1999 12:00 AM




| Element | Weight% | Atomic% |
|---|---|---|
| C K | 2.55 | 4.41 |
| O K | 46.96 | 61.04 |
| Al K | 39.20 | 30.21 |




Comment:

# Project 1

4/2/2013 2:39:08 PM

Spectrum processing :

Peaks possibly omitted : 4.515, 7.465 keV

Processing option : All elements analyzed (Normalised)

Number of iterations = 4

Standard :

C CaCO3 1-Jun-1999 12:00 AM

O SiO2 1-Jun-1999 12:00 AM

Al Al2O3 1-Jun-1999 12:00 AM

Si SiO2 1-Jun-1999 12:00 AM

S FeS2 1-Jun-1999 12:00 AM

Ca Wollastonite 1-Jun-1999 12:00 AM

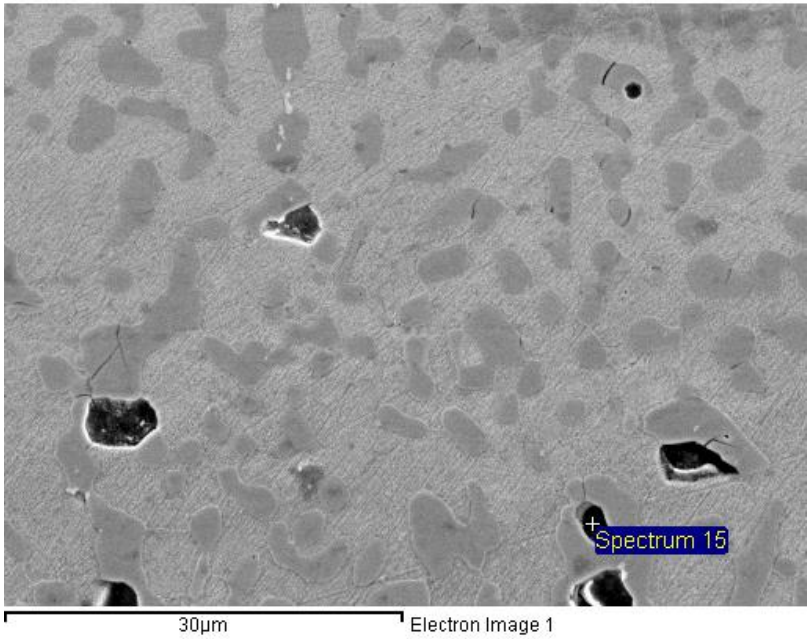
Cr Cr 1-Jun-1999 12:00 AM

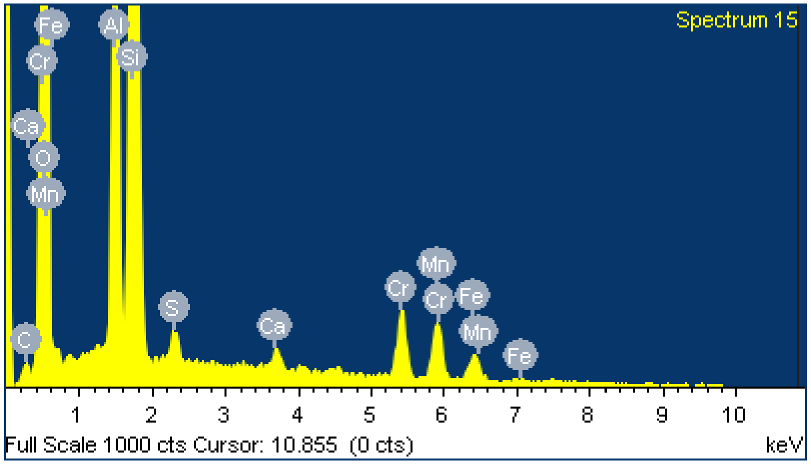
Mn Mn 1-Jun-1999 12:00 AM

Fe Fe 1-Jun-1999 12:00 AM

| Element | Weight% | Atomic% |
|---|---|---|
| C K | 1.94 | 3.36 |

Comment:



Spectrum processing :

No peaks omitted

Processing option : All elements analyzed (Normalised)

Number of iterations = 4

Standard :

C CaCO3 1-Jun-1999 12:00 AM

O SiO2 1-Jun-1999 12:00 AM

Al Al2O3 1-Jun-1999 12:00 AM

Si SiO2 1-Jun-1999 12:00 AM

S FeS2 1-Jun-1999 12:00 AM

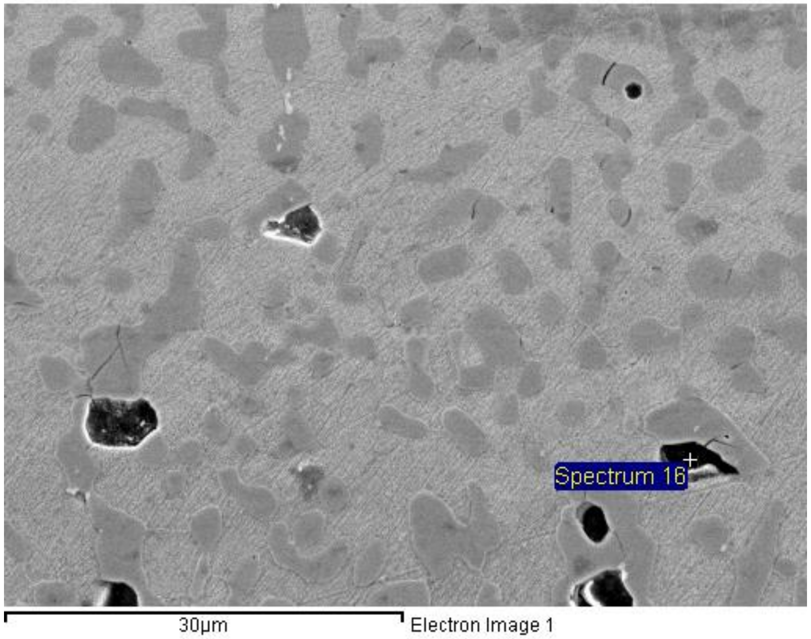
Cr Cr 1-Jun-1999 12:00 AM

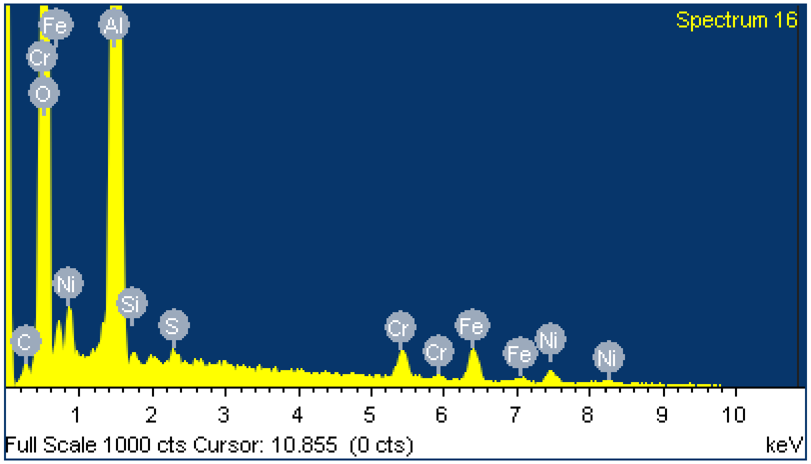
Fe Fe 1-Jun-1999 12:00 AM

Ni Ni 1-Jun-1999 12:00 AM

| Element | Weight% | Atomic% |
|---|---|---|
| C K | 1.70 | 2.87 |
| O K | 50.28 | 63.95 |

Comment:



# Project 1

4/2/2013 2:41:00 PM

Spectrum processing :

No peaks omitted

Processing option : All elements analyzed (Normalised)

Number of iterations = 4

Standard :

C CaCO3 1-Jun-1999 12:00 AM

O SiO2 1-Jun-1999 12:00 AM

Al Al2O3 1-Jun-1999 12:00 AM

Si SiO2 1-Jun-1999 12:00 AM

S FeS2 1-Jun-1999 12:00 AM

Cr Cr 1-Jun-1999 12:00 AM

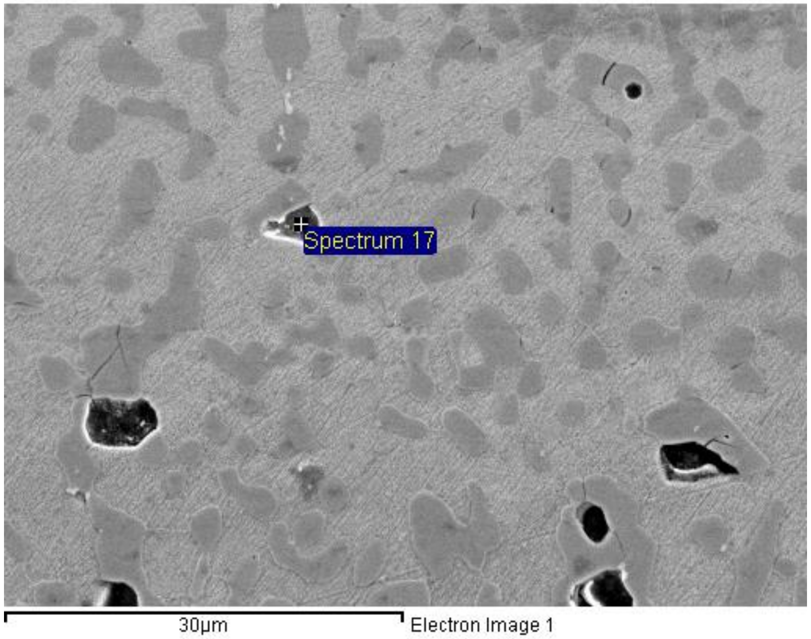
Fe Fe 1-Jun-1999 12:00 AM

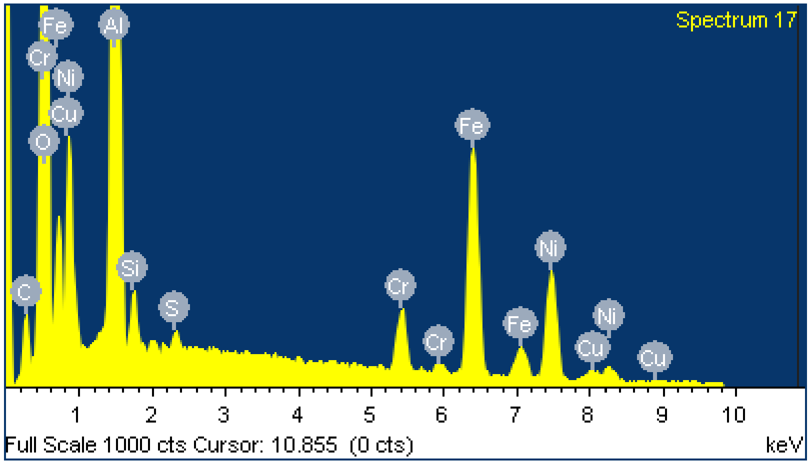
Ni Ni 1-Jun-1999 12:00 AM

Cu Cu 1-Jun-1999 12:00 AM

| Element | Weight% | Atomic% |
|---|---|---|
| C K | 4.76 | 9.79 |

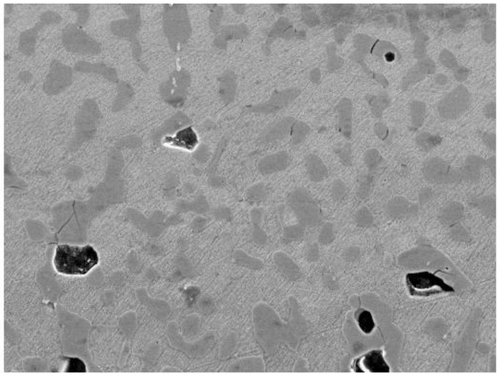
Comment:



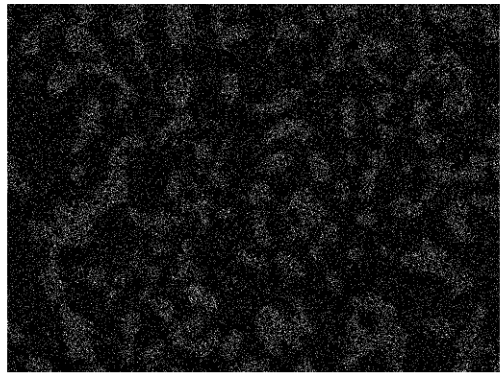



# Project 1

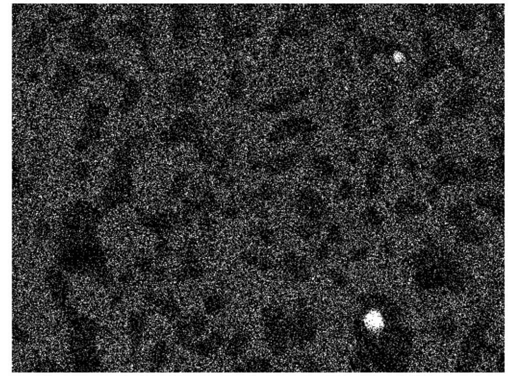
4/2/2013 3:00:48 PM



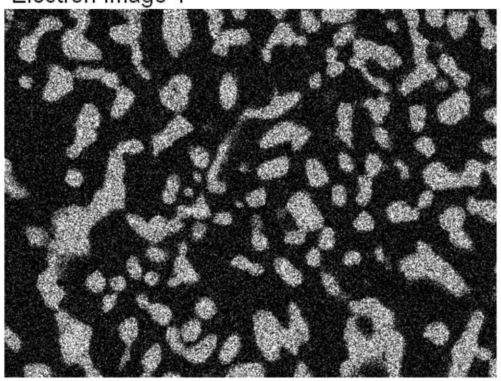
Electron Image 1



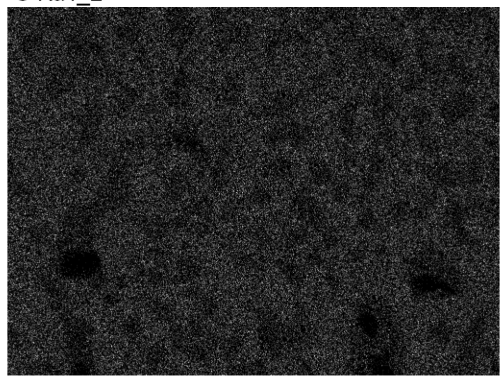
C Ka1_2



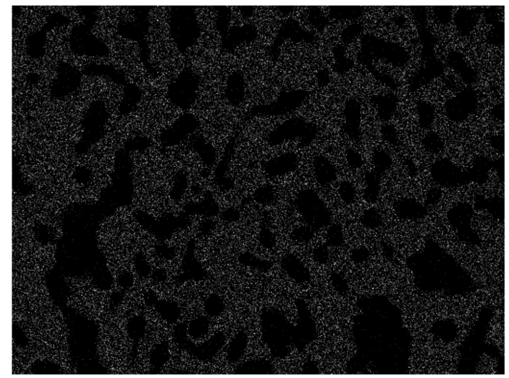
Si Ka1



Cr Ka1



Fe Ka1



Ni Ka1

Cu Ka1

Comment:

## 9.0 Appendix D-OES Results

Three different tests were done on the OES, the following is the raw data from each of the three separate tests completed on the unknown racking post sample.

Program: Fe-01-M
Comment: Orientation -M
Single spark(s)

126319/08

04/03/2013 10:17:33 AM

Elements: Concentration

Sample No:
Sample Id:

Quality:

| No | C<br>% | Si<br>% | Mn<br>% | P<br>% | S<br>% | Cr<br>% | Ni<br>% | Mo<br>% | Al<br>% |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 0.269 | 1.88 | 0.348 | 0.039 | 0.027 | 17.69 | 32.76 | 0.293 | 0.0045 |
| 2 | 1.86 | 1.99 | 0.317 | 0.028 | 0.022 | 16.53 | 33.83 | 0.292 | 0.0037 |
| 3 | 1.84 | 1.98 | 0.318 | 0.032 | 0.021 | 16.67 | 33.62 | 0.290 | 0.0037 |
| 4 | 2.47 | 2.03 | 0.299 | 0.037 | 0.013 | 16.28 | 33.91 | 0.286 | 0.0037 |
| 5 | 2.58 | 1.99 | 0.299 | 0.031 | 0.013 | 16.20 | 33.88 | 0.287 | 0.0044 |

| No | Cu<br>% | Co<br>% | Ti<br>% | Nb<br>% | V<br>% | W<br>% | Pb<br>% | Mg<br>% | B<br>% |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 0.150 | 0.214 | 0.011 | 0.188 | 0.040 | 0.122 | 0.021 | 0.022 | 0.0020 |
| 2 | 0.150 | 0.208 | 0.0093 | 0.201 | 0.032 | 0.107 | 0.011 | 0.022 | 0.0015 |
| 3 | 0.150 | 0.208 | 0.0094 | 0.195 | 0.032 | 0.108 | 0.014 | 0.022 | 0.0015 |
| 4 | 0.245 | 0.206 | 0.0087 | 0.176 | 0.030 | 0.105 | 0.0087 | 0.022 | 0.0013 |
| 5 | 0.251 | 0.205 | 0.0087 | 0.183 | 0.030 | 0.103 | 0.0084 | 0.021 | 0.0012 |

| No | Sb<br>% | Sn<br>% | Zn<br>% | As<br>% | Bi<br>% | Ta<br>% | Ca<br>% | Ce<br>% | Zr<br>% |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 0.0078 | 0.0081 | 0.027 | 0.010 | 0.016 | 0.129 | 0.0008 | 0.019 | 0.011 |
| 2 | 0.0067 | 0.0077 | 0.027 | 0.0082 | 0.017 | 0.064 | 0.0006 | 0.013 | 0.0081 |
| 3 | 0.0062 | 0.0078 | 0.026 | 0.0083 | 0.017 | 0.078 | 0.0006 | 0.014 | 0.0085 |
| 4 | 0.0069 | 0.0075 | 0.026 | 0.0081 | 0.017 | 0.058 | 0.0010 | 0.012 | 0.0075 |
| 5 | 0.0070 | 0.0073 | 0.027 | 0.0062 | 0.017 | 0.049 | 0.0009 | 0.011 | 0.0075 |

| No | La<br>% | Se<br>% | N<br>% | Fe<br>% |
|---|---|---|---|---|
| 1 | 0.0044 | 0.025 | 0.055 | 45.61 |
| 2 | 0.0054 | 0.011 | 0.066 | 44.14 |
| 3 | 0.0050 | 0.014 | 0.067 | 44.23 |
| 4 | 0.0052 | 0.0099 | 0.026 | 43.68 |
| 5 | 0.0056 | 0.0087 | 0.024 | 43.73 |

Program: Fe-30-M 04/03/2013 11:14:04 AM
Comment: Cr-Cr/Ni-steel -M 126319/08
Single spark(s) Elements: Concentration

Sample No: Quality:
Sample Id:

| No | C | Si | Mn | P | S | Cr | Ni | Mo | Al |
|---|---|---|---|---|---|---|---|---|---|
| | % | % | % | % | % | % | % | % | % |
| 1 | 2.11 | 2.07 | 0.277 | 0.024 | 0.017 | 16.82 | 35.28 | 0.226 | 0.0041 |
| 2 | 1.27 | 2.04 | 0.296 | 0.027 | 0.017 | 17.17 | 34.93 | 0.223 | 0.0041 |
| 3 | 1.41 | 2.05 | 0.293 | 0.027 | 0.015 | 17.07 | 34.96 | 0.220 | 0.0041 |
| 4 | 0.259 | 1.90 | 0.311 | 0.022 | 0.023 | 17.84 | 33.89 | 0.223 | 0.0043 |
| 5 | 0.275 | 1.98 | 0.319 | 0.026 | 0.018 | 17.96 | 34.06 | 0.229 | 0.0045 |

| No | Cu | Co | Ti | Nb | V | W | Pb | B | Sb |
|---|---|---|---|---|---|---|---|---|---|
| | % | % | % | % | % | % | % | % | % |
| 1 | 0.284 | 0.196 | 0.0097 | 0.216 | 0.030 | 0.090 | 0.0026 | 0.0015 | 0.0046 |
| 2 | 0.171 | 0.201 | 0.011 | 0.213 | 0.033 | 0.093 | 0.0073 | 0.0017 | 0.0039 |
| 3 | 0.169 | 0.200 | 0.011 | 0.210 | 0.032 | 0.092 | 0.0070 | 0.0018 | 0.0042 |
| 4 | 0.170 | 0.211 | 0.012 | 0.187 | 0.038 | 0.101 | 0.0098 | 0.0019 | 0.0049 |
| 5 | 0.173 | 0.210 | 0.012 | 0.203 | 0.038 | 0.102 | 0.0091 | 0.0018 | 0.0068 |

| No | Sn | As | Bi | Ta | Ca | Se | N | Fe |
|---|---|---|---|---|---|---|---|---|
| | % | % | % | % | % | % | % | % |
| 1 | 0.0050 | 0.012 | >0.0072 | <0.010 | 0.0017 | 0.018 | 0.014 | 42.27 |
| 2 | 0.0054 | 0.014 | >0.0072 | 0.049 | 0.0004 | 0.032 | 0.057 | 43.11 |
| 3 | 0.0054 | 0.014 | >0.0072 | 0.047 | 0.0003 | 0.031 | 0.049 | 43.05 |
| 4 | 0.0055 | 0.013 | >0.0072 | 0.083 | 0.0005 | 0.040 | 0.030 | 44.61 |
| 5 | 0.0053 | 0.016 | >0.0072 | 0.067 | 0.0007 | 0.035 | 0.031 | 44.20 |

Program: Fe-01-M | 04/03/2013 10:34:26 AM
Comment: Orientation -M | 126319/08
Single spark(s) | Elements: Concentration

Sample No: | Quality:
Sample Id:

| No | C | Si | Mn | P | S | Cr | Ni | Mo | Al |
|---|---|---|---|---|---|---|---|---|---|
| | % | % | % | % | % | % | % | % | % |
| 1 | >1.68 | 2.41 | 0.342 | 0.057 | 0.039 | 16.55 | 34.30 | 0.306 | 0.0085 |
| 2 | 0.286 | 1.92 | 0.356 | 0.040 | 0.027 | 17.86 | 32.55 | 0.297 | 0.0039 |

| No | Cu | Co | Ti | Nb | V | W | Pb | Mg | B |
|---|---|---|---|---|---|---|---|---|---|
| | % | % | % | % | % | % | % | % | % |
| 1 | 0.147 | 0.153 | 0.0097 | 0.251 | 0.032 | 0.115 | 0.016 | 0.023 | 0.012 |
| 2 | 0.157 | 0.216 | 0.011 | 0.194 | 0.040 | 0.117 | 0.021 | 0.022 | 0.0020 |

| No | Sb | Sn | Zn | As | Bi | Ta | Ca | Ce | Zr |
|---|---|---|---|---|---|---|---|---|---|
| | % | % | % | % | % | % | % | % | % |
| 1 | 0.0061 | 0.0070 | 0.026 | <0.0010 | 0.019 | 0.057 | 0.0037 | 0.015 | 0.0071 |
| 2 | 0.0079 | 0.0081 | 0.026 | 0.0099 | 0.016 | 0.131 | 0.0007 | 0.020 | 0.011 |

| No | La | Se | N | Fe |
|---|---|---|---|---|
| | % | % | % | % |
| 1 | 0.0052 | 0.013 | 0.046 | <37.39 |
| 2 | 0.0047 | 0.023 | 0.051 | 45.58 |